# The Journal of the

# SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 6

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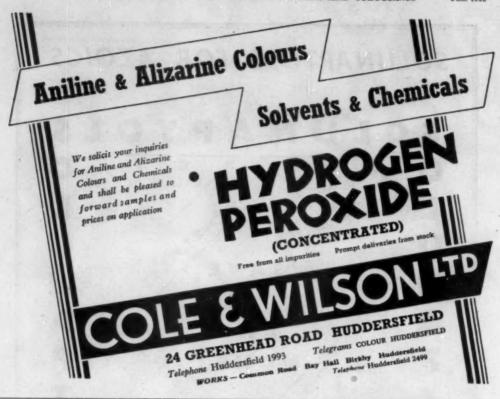
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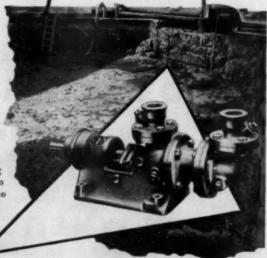
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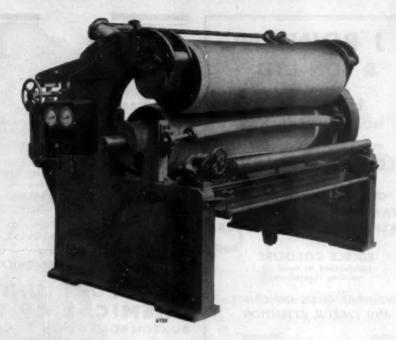
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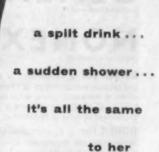
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#### NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1955 and pages 262-266 of the July 1954 issues of the Journal, or write to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). Editorial Communications should be addressed to The Editor, at the same address.

#### Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the Journal—

#### LECTURES

- The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°c. A. S. Fern
- The Dyeing of Textile Fibres at Temperatures above 100°c. 7. A. Fowler
- The Fixation of Direct Cotton Dyes R. Gill
- Textile Printing in London and the Home Counties

  D. King
- Some Observations on the Dyeing of Wool at High Temperatures
  - D. R. Lemin, H. Sagar, and G. A. Coutie

#### COMMUNICATIONS

- The Reflectance of Dyed Fabrics in relation to Dye Concentration
  - E. Atherton

The Polarography of Azo Dyes

J. de O. Cabral and H. A. Turner

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H. R. Richards and J. B. Speakman

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#### THE JOURNAL

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## Society of Dyers and Colourists

Volume 71 Number 6

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#### Proceedings of the Society

#### The Dyeing of Mixtures of Terylene Polyester Fibre and Wool

A. S. FERN and H. R. HADFIELD

Meeting of the Huddersfield Section held at the Co-operative Society Café, Huddersfield, on 19th October 1954, Mr. J. Calvert in the chair

This paper describes an investigation of the dyeing properties of Terylene—wool mixtures. Details are given of the dyeing variables which may affect the partition of dyes between Terylene and wool, and results are presented which enable the most suitable disperse dyes to be selected. A comparison has been made of the fastness properties of laboratory-dyed Terylene—wool mixture material with those of commercially piecedyed wool. A suggested practical dyeing method for Terylene—wool mixtures is given.

The search for fibre mixtures which will have desirable characteristics, often different from those of the individual constituents, is very active. In particular, interest is being shown in blends of Terylene polyester fibre with wool, on which the dyer will inevitably be required to achieve a variety of coloured effects having well defined fastness properties. It is the purpose of this paper, not to discuss the merits of such Terylene blends, but to indicate how far the existing dyeing methods for polyester fibres are applicable when wool is present.

The most satisfactory method at present of obtaining either solid shades or contrast effects on Terylene-wool blends is to manufacture these from the individual components dyed separately in the best way. Thus the wool could be dyed, as loose stock or slubbing, with acid milling, chrome, or metal-complex dyes, and the Terylene loose stock or slubbing with disperse or azoic dyes 1.2. One of the main properties of Terylene-wool blends is their ability, provided the proportion of Terylene is sufficiently high, to be durably pleated by a steaming treatment. Dyes for the Terylene should normally be selected from those possessing the best fastness to sublimation and migration in steam, and the following are recommended—

DISPERSE DYES
DISPERSO FAST YELLOW A
DISPERSO FAST YELLOW GR
DISPERSO FAST Orange B
DURANOL BRILLIAM VIOLET BR
DURANOL BRILLIAM VIOLET BR
DURANOL BRUE G
DURANOL BLUE GN

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Brentosyn BB with Brentamine Fast Red B, Red 2G, Red 3GL, and Blue B Bases, and with Dispersol Fast Orange G and Dispersol Diazo Black B Brentosyn RB with Dispersol Diazo Black B.

It is usually necessary to apply the above dyes in the presence of Tumescal D (ICI) or o-phenylphenol (2-hydroxydiphenyl), or at a temperature of at least 120°c., if medium or heavy dyeings are required.

The dyeing of wool loose stock or slubbing to match the colours and fastness properties obtainable from the above selection is a well established process.

Although this method allows the production of Terylene-wool blended yarns with the best fastness properties, it suffers from a lack of flexibility. Manufacturers frequently prefer to hold stocks of grey blended yarns or fabrics which can be dyed, when required, to mode shades. It is with this problem that the present paper is concerned.

General considerations and preliminary experiments emphasised the importance of the following characteristics of the individual fibres in Terylene-wool mixtures—

- (1) The staining of wool by all the disperse dyes applied at 100°c. was far too great, in absence of a carrier, to allow the production of a range of reasonably fast solid dyeings. Scouring in Lissapol NC (ICI) after dyeing was advantageous, as it removed a large proportion of the disperse dye stain from the wool.
- (2) The staining of wool in a Terylene-wool mixture dyed without a carrier is much greater

than that found in mixtures of nylon or acetate rayon and wool. It is thought that this heavy staining is connected with the slow rate of diffusion of disperse dyes into Terylene compared with nylon or acetate rayon. On 100% Terylene fibre the difficulties caused by the low diffusion rates of disperse dyes can be overcome either by dyeing with a carrier or by dyeing at 120–130°c. At such temperatures wool would, however, suffer considerable damage 3.

(3) Unless the loose disperse dye stain can be removed from the wool at the completion of dyeing, the fastness of the dyed mixture to light, washing, perspiration, and rubbing will be seriously reduced.

(4) An important stage in the production of fast medium and heavy dyeings on Terylene is a "reduction-clearing" process, involving treatment in a solution of sodium hydroxide, sodium hydrosulphite, and Lissolamine A 50% (ICI) at 60°c.2. This treatment improves the rubbing fastness of the dyeing by removing loose dye from the fibre surface. Wool will not withstand this treatment without serious damage.

(5) In the azoic process for dyeing Terylene an essential step consists in treating the material with sodium nitrite and hydrochloric acid at 85°c. for 20 min. Wool is seriously affected by this treatment, so that, at present, there is no possibility of applying azoic dyes to the Terylene component of a Terylene-wool mixture.

(6) Acid, chrome, and metal-complex dyes completely reserve, or only very slightly stain, Terylene. Provided the dyes for the wool are stable under the dyeing conditions found necessary for the Terylene component, no special difficulties will be encountered in dyeing the wool in these mixtures.

With these six factors in mind, the following limitations were accepted in investigating this problem more closely—

- (a) Dyeing at 100°c. in presence of a carrier
- (b) Avoiding an alkaline reduction-clearing treatment
- (c) Using disperse dyes, not azoic combinations
- (d) Ensuring that dyes used for the wool are not affected by the Terylene dyeing process.

#### Experimental

#### 1. MATERIALS

TERYLENE POLYESTER FIBRE—2/28s Hosierytwist staple yarn.

Wool-2/32s, 60s Botany worsted yarn.

MIXTURE MATERIAL—50:50 Terylene-worsted plain-weave fabric (warp and weft blended yarns).

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#### 2. SCOURING

All yarns and fabrics were scoured before dyeing in a liquor containing—

1 part Ammonia liquor (sp.gr. 0-880) per 1000 parts 2 parts Lissapol N (ICI) at 40°c, for 15 min., and then thoroughly rinsed in cold water. Unless otherwise stated, the material was scoured after dyeing, for 20 min. at 50°c, in a bath containing 2 parts of Lissapol NC (ICI) per 1000 parts of liquor.

#### 3. DYEING

Unless otherwise stated, a liquor: goods ratio of 30:1 was used. Lissapol C (ICI) (1 part per 1000 parts of liquor) was the dispersing agent. The sodium salt of o-phenylphenol was obtained by dissolving o-phenylphenol (10 parts) in sodium hydroxide (2-6 parts of flake in 20 parts of boiling water).

#### 4. ASSESSMENT OF STAINING

The Geometric Grey Scales were found to be unsatisfactory for assessing the degree of staining of wool by disperse dyes in presence of Terylene, since the depth of stain may be greater than, equal to, or less than, the depth of the Terylene dyeing. The following arbitrary scale was employed—

- 5. Excellent contrast-wool almost unstained
- 4. Good contrast-wool slightly stained
- 3. Minimum working contrast for medium-depth solid dyeings
- Wool stained and Terylene dyed to approximately the same depth
- 1. Wool stained deeper than the Terylene is dyed.

#### 5. FASTNESS TESTS

These were carried out according to the ICI manual 4.

TABLE I

#### Effect of o-Phenylphenol on Staining of Wool by Disperse Dyes (Arbitrary scale)

Dye (0-5% depth)	No Carrier	o-Phenylphenol (3 g./litre)
Dispersol Fast Yellow G 300	1	2
Dispersol Fast Yellow GR 300	1	. 3
Dispersol Fast Orange G 300	2	3
Dispersol Fast Scarlet B 150	1	4
Duranol Red GN 300	4	5
Dispersol Fast Crimson B 300	2	4
Duranol Red 2B 300	3	5
Duranol Red X3B 300	1	4
Duranol Violet 2R 300	2	4
Duranol Blue GN	3	5

#### TABLE II

#### Effect of Various Carriers on Staining of Wool by Disperse Dyes (Arbitrary scale)

	(Ar	bitrary	y scale)		
Carrier	(g.	onen. /litre)	2% Duran Red 2B 36	ol 00	1% Disperso Fast Orange G 300
o-Phenylphenol		2.5	4		3
p-Phenylphenol		2.5	4		3
Diphenyl	000	2	4		. 3
Naphthalene		2	4-5		3-4
Monochlorobenzene	***	3.3	4-5		3
o-Dichlorobenzene		3.3	2		. 3
Benzoic acid		30	2		1
Salicylic acid		30	2		1
Diphenylamine		2-7	3		3
Fluorene		2	5		3-4
Tetrahydronanhtha	lone	9.7	4		9.4

#### Results and Discussion

### A. PARTITION OF DISPERSE DYES BETWEEN TERYLENE AND WOOL

(a) Effect of a Carrier

Table I shows the results of dyeing a small range of Duranol (ICI) and Dispersol (ICI) dyes without a carrier and in presence of o-phenylphenol. The staining of the wool was assessed according to the scheme outlined. In all cases the carrier significantly increased the proportion of dye absorbed by the Terylene, and decreased the staining of wool. This may be attributed to the increased rate of diffusion of disperse dye into Terylene which occurs when a carrier is employed.

(b) Choice of Carrier

Many organic substances known to be effective carriers for Terylene were examined for their influence on the partition of disperse dyes between Terylene and wool. The results for two dyes are shown in Table II. o-Dichlorobenzene, benzoic acid, and salicylic acid were inferior to o-phenylphenol in reducing the staining of wool. Naphthalene and fluorene were slightly superior to o-phenylphenol, but there are drawbacks to the practical use of these compounds as carriers, owing to their volatility and smell; in addition, according to unpublished work by H. R. Hadfield, fluorene has a markedly detrimental effect on the light fastness of disperse dyes on Terylene. It was decided, therefore, to use o-phenylphenol as carrier throughout, owing to its comparative suitability under bulk conditions.

#### (c) Dyeing at Temperatures above 100°0.

It is known <sup>2</sup> that dyeing at temperatures above 100°c. increases the rate of absorption of disperse dyes by Terylene. An increase in dyeing temperature might therefore be thought to confer advantages similar to those given by a carrier at 100°c. Where wool is present, the temperature cannot be raised above about 110°c. without the danger of severe degradation. Table III shows the effect of dyeing at 110°c. on the partition of disperse dyes between Terylene and wool.

TABLE III

#### Effect of Dyeing above 100°C, on Staining of Wool by Disperse Dyes (Dyeing time 1 hr.) (Arbitrary scale)

Conen. of o-phenylphenol (g./litro)	0	3	0
Temp. of dyeing	98°0.	- 98°c.	110°c.
2.0% Dispersol Fast Yellow G 300	1	2	3
1.0% Dispersol Fast Orange G 300	2	3	2
2-0% Duranol Red GN 300	2	. 5	5
2-0% Duranol Red 2B 300	2	4	4
1.5% Duranol Violet 2R 300	1	4	3
1.5% Duranol Blue GN	2	4	4

Although the staining of wool was less at 110°c. than at 98°c. in absence of carrier, it was no less

than that obtained at 98°c. with o-phenylphenol present. In view of the uncertain position concerning the processing of wool in bulk at 110°c., and the relatively small advantage to be gained by dyeing Terylene at such a temperature (in absence of carrier), attention was temporarily withdrawn from this line of approach.

(d) Effect of Dyebath pH

It has been reported that the acidity of the dyebath affects the partition of disperse dye between acetate rayon and wool. A series of dyeings on Terylene-wool mixtures was carried out with Dispersol Fast Yellow A, Dispersol Fast Orange G, and Duranol Blue G, the pH of the dyebath being varied between 3 and 8-5 and o-phenylphenol being used as carrier. Although slight differences in partition between the Terylene and the wool were observed, and staining of the wool tended to be heavier at pH 3 than at pH 7, these differences were not of sufficient magnitude, with the dyes examined, to be of any practical significance.

TABLE IV

## Staining of Wool by Disperse Dyes (Dyed in presence of o-phenylphenol (3g./litro))

		(Arbitrary scale)		
	2%	Dispersol Fast Yellow A 300		3
ì	2%	Dispersol Fast Yellow G 300		2
	2%	Dispersol Fast Yellow GR 300		3
	2%	Dispersol Yellow 3G 300	000	2
	2%	Duranol Brilliant Yellow 6G 300		4
	4%	Dispersol Fast Orange A 150	000	4
	2%	Dispersol Fast Orange B 300		2
	1%	Dispersol Fast Orange G 300		3
	2%	Duranol Orange G 300		5
	2%	Dispersol Fast Orange Brown R	N	
		150	**	2
	1.5%	Dispersol Fast Crimson B 150	00	3
	6.0%	Dispersol Fast Red R 300		1
	1.5%	Dispersol Fast Scarlet B 150	0.0	2
	2%	Duranol Red 2B 300		4
	2%	Duranol Red GN 300		8
	2%	Duranol Red X3B 300		3
	1.5%	Duranol Brilliant Violet BR 300.		2
	1.5%	Duranol Violet RN 300		2
	1.5%	Duranol Violet 2R 300		4
	2%	Duranol Blue G 300		4
	1.5%	Duranol Blue GN		4
	1.5%	Duranol Blue 2G 300		3
	4%	Duranol Brilliant Blue BN 300 .		4
	3%	Duranol Brilliant Blue CB 300 .		3
	3%	Duranol Brilliant Blue G 300 .		4
	1.5%	Duranol Blue Green B 300 .		1

(e) Effect of Depth of Dyeing

Tests on a number of disperse dyes showed that, in every case, as the amount of disperse dye used was increased, staining of the wool component was increased. The difficulty of producing fast heavy dyeings is undoubtedly associated with this behaviour.

After this preliminary examination of dyeing variables, a general method for dyeing Terylene-wool blends emerged, comprising dyeing at 100°c. in presence of o-phenylphenol. A wide range of Duranol and Dispersol dyes were examined in

this way, and the staining assessments, for medium depths, are given in Table IV.

Dyes which may be used satisfactorily, as far as their partition properties are concerned, are those whose staining of wool is rated at 3 or higher.

#### B. EFFECT OF WOOL-DYEING ASSISTANTS ON BEHAVIOUR OF DISPERSE DYES

Two processes may be used for dyeing the wool component of Terylene blends. In the first, acid, metal-complex, or chrome dyes are applied at the same time as disperse dyes. In the second, the Terylene is dyed in one bath and the wool filled in from a second bath. A similar method has been described by Turnbull 7. If the single-bath method is used, it is desirable to have information on the effect of wool-dyeing assistants on the behaviour of the disperse dyes used.

Dyebaths normally used for applying acid levelling, acid milling, metal-complex, and chrome dyes were found to have no adverse effect on (a) the partition of a wide range of disperse dyes between Terylene and wool, and (b) the physical condition of aqueous dispersions of Duranol and Dispersol dyes recommended for use on Terylene.

In the chroming process, however, the stain caused by certain disperse dyes on wool differed in colour from the stain produced under normal dyeing conditions, owing to combination of dye with the chrome mordant. This effect was more noticeable as the dyebath acidity increased. Thus Duranol Red 2B and Duranol Red X3B are considered unsuitable for application to Terylene-wool mixtures by this method. As these are the preferred red components for Terylene, it will be seen that the use of the chrome dyeing technique is limited.

#### C. EFFECT OF 0-PHENYLPHENOL ON THE FASTNESS OF WOOL DYES

o-Phenylphenol can, if allowed to remain in Terylene fibre dyed with disperse dyes, lower the light fastness in some cases <sup>2</sup>. To determine whether this carrier could cause a similar deterioration in the wool component, seven typical acid milling dyes were applied to Botany wool serge and an equal weight of Terylene staple fabric, in absence and in presence of o-phenylphenol. Under these conditions, o-phenylphenol had no effect on the daylight fastness of the acid dyes examined.

#### D. CLEARING TREATMENTS

The normal "reduction-clearing" treatment recommended for dyed Terylene 2 cannot be given in presence of wool owing to degradation of the wool fibre. Although for pale and medium depths the scouring treatment after dyeing may confer sufficient improvement in rubbing fastness, this type of treatment is not effective enough for heavy dyeings. An oxidation treatment, with potassium permanganate, is conventionally used for clearing disperse dye stains from a variety of fibres in presence of acetate rayon. This process has been modified for Terylene-wool blends, and the optimum concentration of potassium permanganate determined from experiments on Terylene-wool fabric dyed to a solid navy blue with (a) disperse and acid milling dyes and (b) disperse and chrome dyes. The results are given in Table V. The detailed conditions for the treatment potassium permanganate are given later.

Qualitative work with a variety of oxidising agents which wool could be expected to withstand also showed that potassium permanganate was more efficient than potassium persulphate, potassium perborate, or potassium percarbonate. From the results in Table V, treatment with

From the results in Table V, treatment with acidic potassium permanganate at a minimum concentration of 0-4 part per 1000 parts liquor at 40°c., followed by a sodium bisulphite treatment (I part of 15% sodium bisulphite liquor per 1000 parts of solution at 40°c.) to remove the brown oxide stain, must be regarded as an essential part of the dyeing process for heavy shades on Terylene—wool. The effect of this treatment on the physical properties of wool has not been investigated in detail, but recent work 8 with considerably greater quantities of potassium permanganate and sodium bisulphite has shown that the degradation is not excessive.

TABLE V

Effect of Clearing Treatments on Terylene-Wool Mixtures dyed Navy Blue

Treatment after Dyeing		Dyeing	F	astness to Wa	itert	Fastness
		Method*	Effect on Dyeing	Stain on Terylene	Stain on Wool	to Dry Rubbing
Rinsed in cold water	•••	(a) (b)	5 5	4 5	1 2	1
Scoured in Lissapol NC (2 g./litre) at 50°c		(a) (b)	5	4–5 5	2 3	1 2
KMnO <sub>4</sub> (0·2 g./litre) and scoured	000	(a) (b)	5 5	4-5 5	3-4	1 2
KMnO <sub>4</sub> (0.4 g./litre) and secured		(a) (b)	5 5	4–5 5	3-4	3 4
$\rm KMnO_4~(0.6~g./litre)$ and secured $\ , \ \ \dots$	000	(a) (b)	5 5	4–5 5	4	3

<sup>• (</sup>a) Disperse plus acid milling dyes

<sup>(</sup>b) Disperse plus chrome dyes

<sup>†</sup> A water test was selected as it was found to be particularly sensitive to the presence of loose dye on the fibre surface,

TABLE VI

Comparison of Fastness Properties of All-wool and Terylene-Wool Fabrics

(Dyed with disperse and acid milling dyes)

Fabrio	Depth of Dyeing	Light Fast- ness	Rubbing (Dry)		rage of Fairation Staining of Wool		stings h at 55°c. Staining of Wool	Alkalin Effect on Dyeing	e Milling Staining of Wool
Commercially dyed 100% wool	Pale Heavy	4 5	5 4	4-5	4-5	3-4	4-5	2 2	3-4
Laboratory-dyed 100% wool	Pale Heavy	6-7	5 4	5 5	3-4	4-5 4-5	4-5 2-3	2 4-5	4 8
Laboratory-dyed 50:50 Terylene-wool	Pale Heavy	6-7	5 3	5 5	5 2	4-5 8	5 2	3 4–5	5 2–3

Where a permanganate clearing treatment is used, dyes for the wool component must be chosen which are least affected by the process. It was found impracticable to select suitable dyes on the basis of a single laboratory test, using a homogeneous dye at an arbitrary depth. It is therefore suggested that any required shade be matched, and then tested for suitability.

#### E. FASTNESS PROPERTIES

Fifteen samples of commercially piece-dyed wool fabric were tested for fastness to light, water, perspiration, washing, alkaline milling, potting, and rubbing. The dyed samples were divided into pale and heavy shades, and the average fastness to each agency was determined (Table VI). For comparison, a grey, fawn, navy, dark brown, and black were dyed on—

- (a) 50:50 Terylene-wool fabric, each shade being matched with disperse and acid milling dyes applied from the same bath.
- (b) 100% wool using the same acid dyes as in (a).

Similar fastness tests were carried out, and the numerical assessments averaged as before (Table VI).

In both pale and heavy shades, the laboratory-dyed Terylene-wool was equal or superior to the commercially dyed worsted in fastness to light, water, perspiration, washing, and alkaline milling. The heavy shades were inferior in fastness to rubbing. These observations do not imply that the dyed Terylene-wool is satisfactorily fast to these agencies, but merely represents a comparison with commercially accepted piece-dyed wool.

With laboratory-dyed wool, the blend was found to be inferior in fastness properties, particularly in heavy dyeings. Conversely, it may be argued that commercial dyeings of Terylene-wool will be somewhat inferior in fastness to laboratory dyeings.

It is thought that the responsibility for poor rubbing fastness may be attributed to two factors—

(1) The Terylene component is not dyed to the maximum fastness possible with the dyes used: loose dye is present which cannot be effectively removed in presence of wool.

(2) Disperse dyes stain the wool, and this stain cannot be efficiently removed without degrading the fibre.

#### F. PRACTICAL DYEING METHODS

On the basis of the experiments described, Table VII has been constructed to indicate the most important properties required for disperse dyes used to dye mixtures of Terylene with wool. Reference to this table shows that the following combination of disperse dyes is generally preferable—

Dispersol Fast Yellow A Dispersol Fast Yellow GR Duranol Red X3B Duranol Blue G

Dyes for the wool component should be selected in the normal manner, bearing in mind the levelling and fastness requirements, and, in heavy shades, the effect of a permanganate clearing treatment.

The established technique of dyeing 100% Terylene with the aid of o-phenylphenol consists in forming the sodium salt and adding this to the dyebath. The carrier, free o-phenylphenol, is then liberated at 100°c. by adding acetic acid gradually. This process cannot be used in presence of wool owing to the high alkalinity and high temperature involved. On the basis of limited bulk experience, the following technique is proposed—

Set the cold dyebath (temperature below 25°c.) with 3 parts of dissolved sodium o-phenylphenoxide and 1 part of Lissapol C (ICI). The sodium o-phenylphenoxide is prepared by boiling 10 parts of o-phenylphenol, 2.6 parts of sodium hydroxide,

TABLE VII
Selection of Dyes for Terylene-Wool Mixtures

Selection of Dyes for	reryiene-	MOOI WI	xtures
Dye	Partition and Dyeing Properties	Fastness to Light	Fastness to Sub- limation
Dispersol Fast Yellow A		0.0	**
Dispersol Fast Yellow GR		99	80
Duranol Brilliant Yellow 60	. **	-	_
Dispersol Fast Orange G			-
Dispersol Fast Orange A			
Duranol Orange G	. 88	-	-
Duranol Red 2B	. **		
Duranol Red X3B		**	**
Duranol Violet 2R			
Duranol Blue G		*	
* W	odarska		

\*\* Good Noze—In assessing the sultability for light fastness, the behaviour of dyes in combination, as well as individual ratings of light fastness, was used as criterion.

and 20-50 parts of water. Neutralise the cold dye liquor to pH 6.7-7.0 with dilute acetic acid. It will be found that 10 parts of o-phenylphenol, prepared as described above, require approx. 14 parts of acetic acid (30%) for neutralisation. If neutralisation is carried out above 54°c. (the m.p. of o-phenylphenol), globules of carrier may be deposited on the fabric, and these may lead to dye spots.

Enter the secured material and slowly warm to 40°c. At this stage the dye liquor should become clear, as the o-phenylphenol is adsorbed uniformly by the material. Check the pH of the liquor at this stage, and correct to pH 6.5-7.0, if necessary, with acetic acid.

Add the disperse and acid dyes separately, followed by any necessary wool-dyeing assistants. Raise to 100°c. in not less than 30 min, and boil for 40 min. At the end of this period shading additions may be made, and 20 min. should be allowed after each such addition. The total dyeing time at 100°c, must not be less than 60 min.

During the initial period of dyeing the disperse dye tends to be absorbed into the wool; subsequently, as dyeing proceeds, the colour on the Terylene component is built up. It is mainly for this reason that the dyeing time at 100°C. cannot be shortened.

If acid levelling dyes are used in place of neutraldyeing acid dyes, it must be remembered that the sodium acetate, formed as a result of the neutralisation of the sodium o-phenylphenoxide, has a strong buffering action, and comparatively large quantities of sulphuric acid will be required to attain the required dyebath pH.

For pale and medium dyeings, scour after dyeing in a bath containing 2 parts of Lissapol NC per 1000 parts of liquor for 20 min. at 50°c.

For heavy dyeings treat the material in a cold 0.4 part containing of potassium permanganate per 1000 parts of liquor. After 10 min. add 0.5 part of concentrated sulphuric acid (well diluted) and then raise the temperature to 40°c. After a further 10 min. at 40°c. add 1 part of sodium bisulphite (15% liquor) to remove the brown stain of manganese oxide. Rinse the material, and scour as described above to conclude the process

The work described in this paper was done with Botany wool. If coarser qualities are used, staining of the wool fibre by disperse dye is reduced; conversely, finer wools will be more heavily stained. Regenerated protein fibres and reclaimed wools are usually stained very heavily by disperse dyes using the technique described, and union dyeing of materials containing these fibres blended with Terylene is not recommended at present.

#### Note added in Proof

Since this paper was submitted for publication there has been an opportunity to carry out a large series of bulk dyeing trials on Terylene-wool cloth using the method suggested above with o-phenyl-phenol as carrier. These trials have shown that it is extremely difficult to use o-phenylphenol in bulk on Terylene-wool cloth, although satisfactory results are being obtained on blended yarns in circulating machinery. When piece goods are dyed in the winch, the o-phenylphenol must be added as the sodium salt and neutralised with acetic acid in presence of Lissapol C at a low temperature before the fabric is entered; if it were neutralised at 100°c. during dyeing, as for 100% Terylene, the wool would dissolve. Even when the free phenol is liberated as a fine dispersion and the temperature carefully raised to 100°c, after the material has been entered, there is a tendency for unabsorbed carrier to aggregate during the change of phase which occurs at the melting point, 54°c. This may lead to tarry specks of carrier and heavily dyed spots on the cloth.

p-Phenylphenol, on the other hand, with a much higher melting point than the ortho analogue, can be used successfully under bulk conditions to give dyeings free from carrier or dye spots. It is added as an aqueous dispersion, and the only drawback to its use is the present lack of a complete range of suitable disperse dyes with which it can be used. For example, the light fastness of Duranol Red X3B, the preferred red component for Terylene-wool, is excessively reduced by p-phenylphenol, and Duranol Red 2B must be used instead, with Dispersol Fast Yellow GR and Duranol Blue G. Unfortunately, Duranol Red 2B lowers the sublimation and pleating fastness of the final dyeing, and the technique based on p-phenylphenol cannot be indiscriminately recommended at present.

It is hoped to publish further details of these recent developments in the near future.

IMPERIAL CHEMICAL INDUSTRIES LTD.

DYESTUTES DIVISION HEXAGON HOUSE MANCHESTER 9

(MS. received 6th November 1954)

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## PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—XV

## Third Report of the Fastness Tests Co-ordinating Committee

#### Foreword

The Fastness Tests Committee of the Society of Dyers and Colourists was appointed originally in 1927 and issued its First Report in 1934, which contained testing methods for light, perspiration, and washing. In 1948 the Second Report appeared containing testing methods for thirty-three agencies subdivided into four fibre groups. A second edition of this report was published in May 1950, and has remained in force until now.

In 1947 the International Organisation for Standardisation was founded, of which Technical Committee 38 was concerned with all matters affecting textiles. At its first meeting in 1948 a Colour Fastness Subcommittee (ISO/TC 38/SC 1) was formed, and this during the subsequent years has endeavoured to replace all the diverse systems of fastness testing in the United Kingdom, on the Continent, and in the U.S.A. by a single unified system employing the best features from each. The principal active bodies within ISO/TC 38/SC 1 have been the American Association of Textile Chemists and Colorists (A.A.T.C.C.), the Europäisch-Continentale Echtheits-Convention or Groupement d'Études Continental-Européen pour la Solidité des Teintures et Impressions (E.C.E.), and the Society of Dyers and Colourists.

In the *Journal* for November 1953 (J.S.D.C., 69, 409) the Fastness Tests Co-ordinating Committee (which replaced the Fastness Tests Committee in 1950) published for information and comment a range of fastness tests which had been tentatively adopted by ISO/TC 38/SC 1; these tests were simultaneously circulated by the British Standards Institution as a Draft British Standard.

At the June 1954 meeting of ISO/TC 38/SC 1 these tentative tests were finally accepted, minor modifications being necessary in some cases. After ratification at the next meeting of ISO/TC 38 these tests will achieve world-wide international validity as ISO Recommendations.

The activities of ISO/TC 38/SC 1 have by no means ended, however, as fourteen other tests have now become ISO Tentative Tests and two more have been circulated for discussion at the meeting in 1956; about half of these tests have been proposed by the U.K., and are essentially modern versions of the original Second Report tests modified in some cases where experience has shown this to be necessary.

The Fastness Tests Co-ordinating Committee feels that the time is now opportune for all the Second Report tests to be withdrawn and wherever possible replaced by the appropriate ISO Tests—either Recommended, Tentative, or Under Discussion. The ISO Recommendations are unlikely to be changed for many years and will shortly be issued as British Standards; the tests in the other two categories may, however, be changed before achieving the status of ISO Recommendations, but until then they should be regarded as officially sponsored by the Society.

In four cases there are no appropriate ISO Tests, and the following action has been taken-

- 1 Washing Test No. 2 (Wool) included in this Report after significant modifications.
- 2 Washing Test No. 4 (Natural and Regenerated Cellulose)—included in this Report after significant modifications.
- 3 Acid Milling-included in this Report after minor modifications.
- 4 Cellulose Ester Bonding—this was withdrawn (see J.S.D.C., 68, 213 (1952)).

There are in addition six ISO tests for agencies which were not covered in the Second Report; these also become officially sponsored by the Society, and are as follows—

Carbonising with Aluminium Chloride
Acid Felting— Mild
Acid Felting— Severe
Bleaching with Sodium Chlorite
Washing at the Boil
Washing in the presence of Sodium Hypochlorite

The main differences between the tests in the Second Report and those in the Third Report are as follows—

- 1 Tests are no longer subdivided according to fibre, but each test is applicable to all fibres which may be subjected to the agency in question.
- 2 The results of every test (light excepted) are assessed by comparison with two grey scales—the Grey Scale for assessing Change in Colour and the Grey Scale for assessing Staining.
- 3 The method of determining washing fastness by the "pass-fail" system involving four wool and four cotton washing tests is abandoned. Seven tests applicable to any fibre or combination of fibres, if appropriate, are given, the results of each being assessed with grey scales. These seven tests cover a wider range of possible washing treatments than the eight earlier tests.

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#### PROPOSED ISO RECOMMENDATIONS

#### Colour Fastness of Textiles: General Principles of Testing

(PROPOSED ISO RECOMMENDATION)

#### 1. Introduction

- 1.1 This standard provides general information about the methods for testing colour fastness of textiles for the guidance of users. The uses and the limitations of the methods are pointed out, several terms are defined, an outline of the form of the methods is given, and the content of each section is discussed. Procedures common to a number of the methods are discussed briefly.
- 1.2 By colour fastness is meant the resistance of the colour of textiles to the different agencies to which textiles may be exposed during manufacture and subsequent use.
- 1.3 The methods may be used not only for assessing colour fastness of textiles, but also for assessing colour fastness of dyes. When so used, the dye is applied to the textile in specified depths of colour by stated procedures and the textile is then tested in the usual way.
- 1.4 Each method is concerned with colour fastness to a single agency. No directions are given for testing fastness to a succession of agencies, as the agencies of interest in a particular case and the order of application will vary.
- 1.5 The conditions in the tests have been chosen to correspond closely to treatments usually employed in manufacture and to conditions of ordinary use. At the same time, they have been kept as simple and reproducible as possible. As it cannot be hoped that the tests will duplicate all the conditions under which textiles are processed or used, the fastness ratings must be interpreted according to the particular needs of each user. They provide, however, a common basis for testing and reporting colour fastness.

#### 2. General Principle

2.1 A specimen of the textile to be tested, with undyed cloth attached if staining is to be assessed, is subjected to the action of the agency in question. The extent of any change in colour and that of any staining of the undyed cloth are assessed and expressed in fastness numbers.

#### 3. Outline of Form of the Methods

3.1 The headings of the principal sections of the methods follow-

Purpose and scope

Principle

Apparatus and reagents

Specimen

Procedure

Report

Notes

#### 4. Purpose and Scope

- 4.1 Under this heading in each method are given the intended use of the method, its limitations, and definitions of any terms that may not be clear.
- 4.2 Details of the principal natural, artificial, and synthetic fibres which can be submitted to each test are given. These lists are by no means exclusive, and any dyed or printed cloth not mentioned in the method (whether manufactured wholly from one fibre or from a mixture of fibres) can be submitted to test. In such cases it is necessary to ascertain and note whether the procedure is likely to cause any alteration in the fibre under test. This applies particularly to all the new synthetic fibres (polyacrylonitrile, pure or copolymer; polyvinyl, pure or copolymer; polyester; etc.) currently being developed, of which any list is always likely to be incomplete.

#### 5. Principle of Individual Methods

5.1 A concise statement of the principle of the method is given to enable the user to decide whether the method is what he is seeking.

#### 6. Apparatus and Reagents

- 6.1 The equipment and supplies required for the test are enumerated.
- 6.2 Test Solutions— Distilled water is to be used for making test solutions. The concentrations of baths are given in millilitres per litre (ml./litre) or grams per litre (g./litre). The qualities of chemicals used are given in each method. For crystalline substances the amount of water of crystallisation is given; and for liquids, the density.

#### 7. Specimen

- 7.1 Directions are given for the preparation of the "specimen" or "composite specimen" to be tested and of the "test-control specimens" to be used.
- 7.2 By specimen is meant the small piece of material, usually taken from a larger sample or lot, that is to be submitted to the test.
- 7.3 By composite specimen is meant the specimen with attached undyed cloth used for assessing staining.
- 7.4 By test-control specimen is meant a specimen of known behaviour used to ensure the carrying out of a test correctly.
- 7.5 Piece Goods— Specimens from woven and knitted fabrics, felts, and other piece goods should be free from creases, so that the treatment they receive may result in uniform action over the whole area.
- 7.6 Yarns—Yarns to be tested may be knitted into fabric, from which a specimen is taken. They may be wound parallel, e.g. on a U-shaped wire frame, and then sewn between the undyed cloths. For dry treatments, close winding on a card is recommended. For certain wet treatments without accompanying undyed material, hanks of yarn may be used, tied at both ends.
- 7.7 Fibres Fibres may be tested by combing and compressing into a flat pad, which may then be sewn between the undyed cloths.

#### 7.8 Undyed Cloth for Staining-

- 7.8.1 The undyed cloth to be used for assessing staining, if not otherwise specified, should be of plain weave, of medium weight, and free from finishes, residual chemicals, and chemically damaged fibres. Cotton and linen should be bleached; other materials should be cleaned to their usual degree of whiteness without bleaching.
- 7.8.2 The fluidity value of the cotton cloth used for staining may not exceed 5 rhes when measured in the standard cuprammonium solution by the method of Clibbens and Geake (*J. Textile Institute*, 19, T 77 (1928)).

#### 8. Conditioning

- 8.1 Special conditioning of specimens usually is not necessary, but specimens, and the undyed cloths used with them, should be neither moist nor very dry.
- 8.2 Tests in which differences in moisture content of the specimen and undyed cloths will influence the results should be in standard condition, i.e. in moisture equilibrium with air having a temperature of  $20 \pm 2^{\circ}$ C. (68  $\pm$  4°F.) and relative humidity of 65  $\pm$  2%.
  - Note—In tropical and subtropical countries the standard condition is in moisture equilibrium with air having a temperature of  $27 \pm 2^{\circ}$ C.  $(81 \pm 4^{\circ}$ F.) and relative humidity of  $65 \pm 2\%$ .

#### 9. Procedure

- 9.1 The series of operations through which the specimen is taken are described including the assessment of colour change and staining of undyed cloth. The use of test-control specimens is called for in some of the methods to ensure the carrying out of the tests correctly.
- 9.2 Tolerances Tolerances are given for numerical values of dimensions, temperatures, and times that are considered critical. If no tolerance is given, the precision of the measurements need be only that to be expected when using common instruments and reasonable care. The precision is further indicated by the number of significant figures in the values given.
- 9.3 Liquor Ratio— By liquor ratio is meant the ratio of the volume of the liquor used in the treatment, expressed in millilitres, to the weight of the specimen or composite specimen (specimen plus undyed material), expressed in grams.
- 9.4 Wetting out—Special care must be taken when wetting specimens that they are uniformly saturated. In particular, when wool or materials containing wool are to be wetted out, it is necessary for them to be thoroughly kneaded by hand, with the flattened end of a glass rod, or with a mechanical device.
- 9.5 Spotting— For tests in which the material is spotted with water or a reagent and the drop worked in with a glass rod, care should be taken not to raise the surface of the material, otherwise there will be a change in the reflection of light and hence in appearance.
- 9.6 Wetting to 100% Increase in Weight— When the material is to be wetted to contain its own weight of liquor, it may be saturated with the liquor and then drawn between two rubber rollers, squeezed by means of a rubber roller on a glass plate, or centrifuged. Wringing by hand does not give uniform wetting.
- 9.7 Temperature— Temperatures are given in degrees centigrade (°c.), usually with a tolerance of plus or minus 2 degrees (± 2°c.). For reliable results, continuous temperature control is essential.

#### 10. Fastness of Dyes

As the fastness of a dye depends on the depth of colour, it is necessary to specify standard depths which are to be used by dye manufacturers. A main range of twenty standard depths covering the whole spectrum has been prepared, whose depth is referred to as I; in addition two supplementary ranges have been prepared, one at twice the depth (referred to as 2) and the other at one-twentyfifth of the depth (referred to as 1/25), which are to be used if additional fastness figures for pale and full colours are required. In all cases the fastness figures corresponding to the main standard depth 1 should be given.

These "standard depths" have been prepared on a matt fabric (wool gabardine) and on a bright fabric (viscose rayon).

These "standard depths" are not subjected to any fastness test but merely indicate at what depth of colour the fastness figures of dye manufacturers have been obtained irrespective of dye or fibre.

#### 11. Assessment of Colour Fastness

- 11.1 Colour fastness is assessed separately with respect to change in colour of the specimen in the test and with respect to staining of undyed materials. Specimens and undyed cloths should have cooled after drying and should have regained their normal moisture content before the assessment.
- 11.2 Colour Fastness with respect to Change in Colour—The change which occurs in a test may be a change in depth of colour, a change in hue, a change in brightness, or any combination of these. Regardless of the character of the change, the assessment is based upon the magnitude of the visual contrast between two coloured areas, one being the original material, and the other the tested specimen. This contrast is compared visually with the contrast represented by the five pairs of neutral colour chips (or swatches of cloth) in the standard grey scale specified for the purpose under Colour Fastness: Grey Scale for assessing Change in Colour. Each step of the scale bears a fastness number between 5 (no contrast) and 1 (a large contrast). The fastness rating of the specimen is that number of the grey scale which corresponds to the contrast between the original and the treated specimens. If the latter lies between two of the controls of the scale, the specimen is given an intermediate rating, e.g. 3-4 or 4-3 (these ratings being identical). A rating of 5 is given only when there is no difference between the tested specimen and the original material.

In assessing the results of tests for colour fastness to light the exposed specimens are compared with the scale of eight standard dyed blue wool cloths, which have been exposed simultaneously. A grey scale is used only indirectly as a means of determining the degree to which fading should be carried out in the test. (See Colour Fastness to Light: Daylight.)

- 11.3 Colour Fastness with respect to Staining—The degree of staining of the undyed cloth either by absorption of dye from the bath or by direct transfer of colour from the specimen is assessed by visual inspection of the side of the undyed cloth which was in contact with the specimen. Where the use of adjacent white (undyed) cloths is required by the method of test, the specimen is placed between them: when printed cloths are being investigated, the composite specimen is so arranged that the printed surface is in direct contact with half of each of the two undyed cloths. The colour of the bath is ignored. The standard Grey Scale for assessing Staining is used. It is specified under Colour Fastness: Grey Scale for assessing Staining. The steps of this scale run from 5 (no contrast), represented by a pair of white chips (or swatches of cloth), to 1 (a large contrast), represented by a white and a grey chip. The contrast between the original undyed cloth and that stained as a result of the test is rated with the aid of the scale in a similar way to that described for change in colour in § 11.2. Staining is assessed without reference to the depth of colour of the material tested. The staining is assessed for each type of undyed material in the test.
- 11.4 Influence of Pretreatments and Aftertreatments— It is well known that certain pretreatments and aftertreatments affect the fastness properties of coloured textiles. Thus any change in a coloured material that takes place as a result of applying a fastness test may not necessarily be due to lack of fastness in the dye itself.

#### 12. Conditions of Viewing and Illumination in assessing Colour Fastness

12.1 In assessing colour fastness, a piece of the original material and the tested specimen, or the undyed and stained cloths, are arranged side by side in the same plane and oriented in the same direction. Two or more layers are used if necessary to avoid effects of other backing on the appearance. The appropriate grey scale is placed nearby in the same plane. The

surrounding field is uniform grey of a brightness slightly less than that of the darkest member of the Grey Scale for assessing Change in Colour. The surfaces to be compared are illuminated by north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source with illumination of 50 foot-candles or more. The light is incident upon the surfaces at approx. 45°, and the direction of viewing is approximately along the perpendicular to the plane of the surfaces.

#### 13. Report

13.1 The information to be supplied in the report of the tests is indicated under this heading in the methods.

#### 14. Notes

- 14.1 Sources of supply of materials called for in the tests, references to the literature, and other information not considered necessary for inclusion in the body of the test method are given under "Notes".
  - The existence of a simple method of checking the colour fastness of a dyed cloth by reference to a standard dyeing is extremely useful for authorities receiving goods in large quantities who wish to determine whether these are comparable with an accepted piece.
- 14.2 Where it is not essential to determine the exact colour fastness rating it may be sufficient to compare the cloth being investigated with a standard dyeing: this method is used when it is necessary to establish only whether the colour fastness of the specimen to a particular agency is greater than, equal to, or less than that of the reference dyeing.

#### Colour Fastness: Grey Scale for assessing Change in Colour

(PROPOSED ISO RECOMMENDATION)

Note—An account of the development of grey scales is given in J.S.D.C., 69, 404 (1953)

#### 1. Purpose and Scope

1.1 This standard describes the Grey Scale for assessing Changes in Colour of textiles in colour fastness tests and its use. A precise colorimetric specification of the Scale is given as a permanent record against which newly prepared working standards and standards that may have changed can be compared (see §4.1).

#### 2. Principle

2.1 The Scale consists of five pairs of grey colour chips (or swatches of grey cloth) each representing a visual difference in colour and contrast. The differences represented, which are determined by the Adams chromatic value formula, and corresponding numerical fastness ratings are approximately as follows—

Colour Differences in N.B.S. Units	Fastness Rating		Colour Differences in N.B.S. Units	Fastness Rating
0	5		6	2
11	2001 98 4	and the state of the state of the	12	1
3	3			
		(See §3.3)		

- 2.2 Fastness rating 5 is represented on the scale by two identical chips (or swatches) mounted side by side, neutral grey in colour and having a reflectance of  $12 \pm 1\%$  (see §3). The colour difference is 0.
- 2.3 Fastness ratings 4 to 1, inclusive, are represented by a "reference" chip (or swatch) identical with those used for Grade 5, paired with similar but lighter neutral grey chips (or swatches), the visual differences in the pairs being in geometric steps of colour difference, or contrast, as shown in the table.
- 2.4 Use of the Scale—Place a part of the original textile and the tested specimen of it side by side in the same plane and oriented in the same direction. Place the grey scale nearby in the same plane. The surrounding field should be uniform grey of a brightness slightly less than that of the darkest member of the grey scale. If necessary to avoid effects of the backing on the appearance of the textiles, use two or more layers of the original textile under both original

and tested specimens. Illuminate the surfaces with north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source with illumination of 50 foot-candles or more. The light should be incident upon the surfaces at approx. 45° and the direction of viewing approx/mately perpendicu/ar to the plane of the surfaces. Compare the visual difference between original and tested textile with the differences represented by the grey scale. The fastness rating of the specimen is that number of the grey scale which corresponds to the contrast between the original and the treated specimens. If the latter lies between two of the contrasts of the scale, the specimen is given an intermediate assessment, e.g. 3-4 or 4-3 (these ratings being identical). A rating of 5 is given only when there is no difference between the tested specimen and the original material.

#### 2.5 Describing Colour Changes in Fastness Tests-

- 2.5.1 In using the grey scale as outlined in §2.4, the character of the change in colour, whether in hue, depth, brightness, or any combination of these, is not rated: the overall difference, or contrast, between original and tested specimens is the basis for the assessment.
- 2.5.2 If, as in rating dyes on textiles, for example, it is desired to record the character of the change in colour of the textile in the test, appropriate qualitative terms may be added to the numerical rating, as illustrated by the following examples:—

Rating	Me	aning
3	Contrast equal to Grade 3 of the grey scale	Change is a loss in depth of colour only
3 redder	Contrast equal to Grade 3 of the grey scale	No significant loss in depth, but colour has become redder
3 weaker, yellower	Contrast equal to Grade 3 of the grey scale	Loss in depth and change in hue have occurred
3 weaker, bluer, duller	Contrast equal to Grade 3 of the grey scale	Loss in depth and change in both hue and brightness have occurred
4-5 redder	Contrast intermediate between 4 and 5 of the grey scale	No significant loss in depth, but colour has has become slightly redder

- 2.5.3 When changes in colour occur in two or three directions, it is considered neither feasible nor necessary to indicate the relative magnitude of each change, either in words (such as "much" or "little") or by number (e.g. 1 = small, 3 = large), or even by putting the larger change first (i.e. "bluer, duller" different from "duller, bluer").
- 2.5.4 When the space available for recording qualitative terms is restricted, as on pattern cards, the following abbreviations may be used—

Bl	-	Bluer	R	=	Redder	W	-	Weaker	D	200	Duller
·G	2002	Greener	Y	-	Yellower	S	=	Stronger	Br	=	Brighter

#### 3. Colorimetric Specification of the Grey Scale

- 3.1 The precise colorimetric specification of the standard grey colours of the scale given in this section is derived from §2.
- 3.2 Actual grey scales used as working standards should approximate to these colours closely, particularly in colour spacing.
- 3.3 The colour differences shall be measured by employing the Adams "chromatic value" formula (Nickerson, D., J. Optical Soc. Amer., 34, 550 (1944))—

Colour difference = 
$$40 \{[0.23 \ \Delta \ V_y]^3 + [\Delta \ (V_x - V_y)]^3 + [0.4 \ \Delta \ (V_x - V_y)]^4\}^{0.5}$$
 N.B.S. units

Actual grey scales used as working standards shall not deviate from the values specified in §2.1 by more than the following amounts—

Grade	Theoretical (N.B.S. units)	Tolerance (N.B.S. units)		Grade	Theoretical (N.B.S. units)	Tolerance (N.B.S. units)
5	0	. 0		2	6-0	±0-5
4	1.5	±0·2		1	12-0	±1·0
3	3-0	+0-2	1.			COLUMN TO SERVICE SERV

#### 4. Note

4.1 Grey scales may be obtained from The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.

#### Colour Fastness: Grey Scale for assessing Staining

(PROPOSED ISO RECOMMENDATION)

NOTE—This scale replaces S.D.C. Grey Scale No. 1. An account of the development of grey scales is given in J.S.D.C., 69, 404 (1953)

#### 1. Purpose and Scope

1.1 This standard describes the Grey Scale for assessing Staining of undyed textiles in colour fastness tests and its use. A precise colorimetric specification of the scale is given as a permanent record against which newly prepared working standards and standards that may have changed can be prepared (see §3).

#### 2. Principle

2.1 The scale consists of one pair of white and four pairs of grey and white colour chips (or swatches of grey and white cloth) each representing a visual difference in colour or contrast. The differences represented, which are determined by the Adams chromatic value formula, and corresponding numerical fastness ratings are as follows—

Colour Differences (N.B.S. units)	Fastness Rating	and the same of	Colour Differences (N.B.S. units)	Fastness Rating
0	5		16	2
4	.4		32	. 1
	3			

- 2.2 Fastness rating 5 is represented on the scale by two identical white chips (or swatches) mounted side by side, having a reflectance of not less than 85%. The colour difference is 0.
- 2.3 Fastness ratings 4 to 1, inclusive, are represented by a reference white chip (or swatch) identical with those used for rating 5, paired with similar but neutral grey chips (or swatches), the visual differences in the pairs being in geometric steps of colour difference, or contrast, as shown in the table.
- Use of the Scale-Place a piece of the unstained undyed textile and the treated piece of it side by side in the same plane and oriented in the same direction. Place the grey scale nearby in the same plane. The surrounding field should be uniform grey of a brightness slightly less than that of the darkest member of the Grey Scale for assessing Change in Colour. If necessary to avoid effects of the backing on the appearance of the textiles, use two or more layers of the unstained undyed textile under both unstained and treated pieces. Illuminate the surfaces with north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source of illumination of 50 foot-candles or more. The light should be incident upon the surfaces at approx. 45°, and the direction of viewing approximately perpendicular to the plane of the surfaces. Compare the visual difference between original undyed and treated pieces with the differences represented by the grey scale. The fastness rating of the specimen is that number of the grey scale which corresponds to the contrast between original and treated pieces. If this lies between two of the contrasts of the scale, the specimen is given an intermediate assessment, e.g. 3-4 or 4-3 (these ratings being identical). A rating of 5 is given only if there is no difference between the treated piece and the original undyed material.

#### 3. Colorimetric Specification of the Grey Scale .

- 3.1 The precise colorimetric specification of the standard grey colours of the scale given in this section is derived from §2.
- 3.2 Actual grey scales used as working standards should approximate to these colours closely, particularly in colour spacing.
- 3.3 The colour differences shall be measured by employing the Adams chromatic value formula (Nickerson, D., J. Optical Soc. Amer., 34, 550 (1944))—

Colour difference = 40 {[0.23  $\Delta V_y]^2 + [\Delta (V_x - V_y)]^3 + [0.4 \Delta (V_z - V_y)]^3$ } 0.5 N.B.S. units

Actual grey scales used as working standards shall not deviate from the values specified in §2.1 by more than the following amounts—

Grade	Theoretical (N.B.S. units)	Tolerances (N.B.S. units)	Grade	Theoretical (N.B.S. units)	Tolerances (N.B.S. units)
5	0 .	0	2	16	±1.0
4	4	±0·3	1	32	±2·0
3	8	+0.5			

#### 4. Note

4.1 Grey scales may be obtained from The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.

#### Colour Fastness to Acid Spotting

(PROPOSED ISO RECOMMENDATION)

Note - This test replaces the tests for Acid Spotting in the Second Report

#### 1. Purpose and Scope

- 1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the action of dilute solutions of organic and mineral acids.
- 1.2 Three tests differing in severity are provided. Any or all may be used depending upon the nature of the fibre.

#### 2. Principle

2.1 Drops of the acid solution are worked into the textile with a glass rod, and the changes in colour of the textile while it is still wet and after drying are assessed with a standard grey scale.

#### 3. Apparatus and Reagents

- 3.1 Pipette or dropper
- 3.2 Glass rod with rounded end
- 3.3 Grey Scale for assessing Change in Colour (see §7.1)
- 3.4 Acetic acid solution containing 300 g. glacial acetic acid per litre
- 3.5 Sulphuric acid solution containing 50 g. concentrated sulphuric acid (sp. gr. 1\*84) per litre
- 3.6 Tartaric acid solution containing 100 g. tartaric acid per litre (especially for acetate rayon, instead of acetic acid).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a piece at least 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

#### 5. Procedure

- 5.1 Spot the specimen with two drops of the appropriate solution at room temperature. Work the drops into the specimen with the glass rod.
- 5.2 Dry the specimen in air at room temperature.
- 5.3 Assess the change in colour with the grey scale (see §7.1).

#### 6. Report

6.1 Report the numerical rating for change in colour for each kind of acid used (and for change in colour when wet if of interest).

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing (especially §9.5)
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Alkali Spotting

(PROPOSED ISO RECOMMENDATION)

NOTE - This test replaces the tests for Alkali Spotting in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the action of dilute alkaline solutions.

#### 2. Principle

2.1 Drops of a solution of sodium carbonate are worked into the textile with a glass rod, and the change in colour is assessed with a standard grey scale.

#### 3. Apparatus and Reagents

- 3.1 Pipette or dropper
- 3.2 Glass rod with rounded end
- 3.3 Grey Scale for assessing Change in Colour (see §7.1)
- 3.4 Sodium carbonate solution containing 100 g. anhydrous sodium carbonate per litre

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a piece at least 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

#### 5. Procedure

- 5.1 Spot the specimen with two drops of the sodium carbonate solution at room temperature. Work the drops into the specimen with the glass rod. Dry the specimen in air at room temperature; brush it to remove sodium carbonate residues.
- 5.2 Assess the change in colour with the grey scale (see §7.1).

#### 6. Report

6.1 Report the numerical rating for change in colour.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing (especially §9.5)
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Bleaching with Hypochlorite

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Hypochlorite in Bleaching in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles in all forms to the action of bleaching baths containing sodium and calcium hypochlorite in concentrations normally used in commercial bleaching. It is applicable principally to natural and regenerated cellulose materials.

#### 2. Principle

2.1 A specimen of the textile is agitated in a solution of sodium hypochlorite, rinsed in water, agitated in hydrogen peroxide solution, rinsed, and dried. The change in colour is assessed with a standard grey scale.

#### 3. Apparatus and Reagents

3.1 Sodium hypochlorite solution containing 2 g. of available chlorine per litre, buffered at pH 11·0  $\pm$  0·2 with 10 g. anhydrous sodium carbonate per litre, temperature 20  $\pm$  2°c.

 $(68 \pm 4^{\circ}F)$ . The weight of solution used to be 50 times that of the specimen. Sodium hypochlorite of the following composition shall be used—

Sodium hypochlorite	NaOCI	14-16%
Sodium chloride	NaCl .	12-17%
Sodium hydroxide	NaOH	2.0% maximum
So.lium carbonate	Na <sub>z</sub> CO <sub>a</sub>	2.0% maximum
Iron	Fe	0-001 % maximum

- 3.2 Hydrogen peroxide solution containing 2.5 ml. of 30% hydrogen peroxide per litre. The weight of solution used to be 50 times the weight of the specimen.
- 3.3 Glass or glazed porcelain container which can be closed, for specimen and bleaching solution (see §5.1).
- 3.4 Grey Scale for assessing Change in Colour.

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, use a specimen at least  $10 \text{ cm.} \times 4 \text{ cm.}$  (4 in.  $\times 1\frac{1}{2}$  in.).
- 4.2 If the textile to be tested is yarn, knit it into a fabric and use a specimen of it at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet at least 10 cm. × 4 cm., and sew the sheet on a piece of unbleached desized cotton cloth to support the fibres.

#### 5. Procedure

- 5.1 Wet out the specimen in distilled water, remove the excess water, and place the specimen in an open or loosely rolled form in the sodium hypochlorite solution (see §3.1). Close the container and keep the specimen in the solution at 20 ± 2°c. (68 ± 4°F.) for 60 min. Avoid exposure to direct sunlight.
- 5.2 Wash the specimen thoroughly in cold running water, and then stir it for 10 min. in the hydrogen peroxide solution at room temperature.
- 5.3 Wash the specimen thoroughly in cold running tap-water, remove the excess water, and dry the specimen in air at a maximum temperature of 60°C. (140°F.).
- 5.4 Assess the change in colour of the specimen with the grey scale (see §7.1).

#### 6. Report

6.1 Report the numerical rating for change in colour.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Bleaching with Peroxide

(PROPOSED ISO RECOMMENDATION)

Note-This test replaces the tests for Peroxide Bleaching in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds except polyamide, in all forms, to the action of bleach baths containing peroxide at concentrations commonly employed in textile processing.

#### 2. Principle

- 2.1 A specimen of the textile in contact with specified undyed cloths is immersed in the bleaching solution, rinsed, and dried, and the change in colour and the staining are assessed with standard grey scales.
- 3. Apparatus and Reagents
  3.1 Test tube, of diameter and length such that the test specimen roll will have a reasonably

snug fit in the tube and be covered by the bleach solution

- Reflux condenser, fitting the test tube, may be used to reduce evaporation from the bleach bath during the test
- 3.3 Bleach bath of the composition given in Table I

Composi	tion of Bleach Bath	and Conditions of	Use	
Starting Bath (per litre of distilled water)	Bath 1 (for Natural and Regenerated Cellulose)	Bath 2 (for Cotton and Linen)	Bath 3 (for Wool and Acetate Rayon)	Bath 4 (for Silk)
Hydrogen peroxide (a), ml.	5	, «	20	20
Sodium peroxide (b), g.		3	-	-
Sodium silicate (c), ml.	5	-	-	5
Sodium pyrophosphate (d), g.	40		5	
Magnesium chloride (e), g.	0-1	-	- 44	0-1
pH, initial value $\pm 0.2(f)$	10-5	11-5	9-3 (g)	10-0
Temperature, °c. ± 2°	90	80	50	70
Temperature, °F. ± 4°	194	176	122	158
Duration of treatment, hr.	1	1 30	2	2
Liquor ratio	30:1	30:1	30:1	30:1

- (a) 30% H<sub>2</sub>O<sub>2</sub> by volume
- (b) 100% Na<sub>2</sub>O<sub>2</sub>
   (c) About 26% SiO<sub>2</sub> and 10% Na<sub>2</sub>O (sp. gr. = 1·32, 35° Bé.) (d) Na<sub>4</sub>P<sub>2</sub>O<sub>2</sub>, 10H<sub>2</sub>O
- (e) MgCl<sub>2</sub>,6H<sub>4</sub>O
- Adjust by addition of NaOH soln, if necessary The pH of the bath at the end of the test must not be less than 9-0. (g)
- Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated in Table II, or as otherwise specified.

TABLE II

If First Piece is-Wool, silk, linen, or viscose rayon Cotton or acetate rayon

Second Piece to he-Cotton Viscose rayon

Grey scales for assessing the change in colour and the staining of the undyed cloths (see §7.1).

#### 4. Specimen

- If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of undyed cloth (see §3.4), and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between two undyed cloths, and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 10 cm. × 4 cm. in size, place the sheet between the two undyed cloths, and sew around the four sides to form a composite specimen.

#### 5. Procedure

- 5.1 Loosely roll the composite specimen in the direction of the long edge, place the 4-cm. roll in the appropriate bleach solution (Table I) in the test tube, and keep it well covered for the time and at the temperature indicated in Table I.
- Remove the specimen, rinse it for 10 min. in cold running tap-water, and squeeze it. Remove the stitching on two long sides and one short side, open the specimen out, and dry at a temperature not exceeding 60°c. (140°F.) with the three parts in contact only at the one line of stitching.
- 5.3 Assess the change in colour of the specimen and the staining of the undyed cloths with the appropriate standard grey scales (see §7.1).

#### 6. Report

Report the bleach solution used, the numerical rating for colour change, and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Carbonising with Aluminium Chloride

(PROPOSED ISO RECOMMENDATION)

NOTE- The Second Report did not contain a test for Carbonising with Aluminium Chloride

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles to the manufacturing operation designed to remove vegetable impurities by treatment with aluminium chloride at high temperatures. It is mainly applicable to wool and textiles containing wool, particularly those containing also acetate rayon or polyamide fibres.

#### 2. Principle

2.1 A specimen containing the aluminium chloride solution is dried, baked, rinsed, and neutralised, and the changes in colour after rinsing, neutralising, and drying are assessed with a standard grey scale.

#### 3. Apparatus and Reagents

- 3.1 Oven for drying specimens in air at  $60 \pm 2^{\circ}$ C. (140  $\pm 4^{\circ}$ F.) and baking in air at 115  $\pm 2^{\circ}$ C. (239  $\pm 4^{\circ}$ F.)
- 3.2 Aluminium chloride solution (sp.gr. 1-037) containing 51-4 g. AlCl<sub>2</sub> per litre
- 3.3 Ammonium hydroxide solution containing 2 ml. of 20% NH<sub>4</sub>OH per litre
- 3.4 Test-control—A dyeing of Alizarin Red W (C.I. 1034) treated with potassium dichromate (see §7.1)
- 3.5 Grey Scale for assessing Change in Colour (see §7.2).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a piece at least 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

#### 5. Procedure

- 5.1 Carry out the operation described in §§5.2-5.4 inclusive with the specimen and with the test-control specimen in parallel in separate baths.
- 5.2 Immerse the specimen in the aluminium chloride solution for 15 min. at room temperature (liquor ratio 20:1). Squeeze it to leave in 80% on its weight of solution.
- 5.3 Dry the specimen by hanging it in an oven for 30 min., or longer if necessary, at  $60 \pm 2^{\circ}$ c. (140  $\pm$  4°F.). Then bake it by heating for 15 min. at 115  $\pm$  2°c. (239  $\pm$  2°F.).
- 5.4 Rinse the specimen for 5 min. in cold running tap-water, and then divide it into two equal parts. Dry one half in air at a temperature not exceeding 60°c. (140°F.).
- 5.5 Agitate the other half for 30 min. at room temperature in the ammonium hydroxide solution (liquor ratio 40:1). Then rinse it for 5 min. in cold running tap-water, and dry it in air at a temperature not exceeding 60°c.
- 5.6 Assess the effect on the test control with the grey scales (see §7.2). If the change in colour is not equal to 4-5 yellower on the appropriate scale, the test has not been carried out correctly, and the operations described in §§5.1-5.5 inclusive must be repeated with a fresh specimen and a fresh test-control specimen.
- 5.7 Assess the change in colour of each half of the specimen with the grey scale (see §7.2).

#### 6. Report

6.1 Report the numerical rating for change in colour for both the rinsed and the neutralised portions of the specimen.

#### 7. Notes

7.1 Test-control—A well wetted-out pattern of wool cloth is entered at 40°C. (104°F.) into a dyebath containing 1% Alizarin Red W powder, 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O), and 3% acetic acid (30%), all percentages being calculated on the weight of the pattern, at a liquor ratio of 40:1.

The dyebath is raised to the boil in 30 min. and boiled for a further 30 min. If necessary, the dyebath is exhausted by careful addition of 1-3% acetic acid (30%) or 1% sulphuric acid (sp.gr. 1-84) well diluted with water. The bath is boiled for a further 15 min. after addition of the acid.

The dyebath is cooled down by addition of cold water, and 0.5% potassium dichromate dissolved in water is added. The dyebath is raised to the boil and boiled for 30 min.

The pattern is then removed, rinsed, and dried.

- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Carbonising with Sulphuric Acid

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Carbonising in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles to the manufacturing operation designed to remove vegetable impurities by treatment with sulphuric acid at high temperatures. It is mainly applicable to wool and textiles containing wool.

#### 2. Principle

- 2.1 A specimen containing the sulphuric acid solution is dried, baked, rinsed, and neutralised, and the changes in colour after rinsing, neutralising, and drying are assessed with a standard grey scale.
  - 3. Apparatus and Reagents
- 3.1 Oven for drying specimens in air at  $60 \pm 2^{\circ}$ C.  $(140 \pm 4^{\circ}$ F.) and baking in air at  $105 \pm 2^{\circ}$ C.  $(221 \pm 4^{\circ}$ F.)
- 3.2 Sulphuric acid solution containing 50 g. concentrated sulphuric acid (sp.gr. 1-84) per litre
- 3.3 Sodium carbonate solution containing 2 g. anhydrous sodium carbonate per litre
- 3.4 Test Control—A dyeing of Alizarin Red W (C.I. 1034) treated with potassium dichromate (see §7.1)
- 3.5 Grey Scale for assessing Change in Colour (see §7.2).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a piece at least 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

#### 5. Procedure

5.1 Carry out the operations described in §§5.2-5.7 inclusive with the composite specimen and the composite test-control specimen in parallel, in separate baths.

- 5.2 Immerse the specimen in the sulphuric acid solution for 15 min. at room temperature (liquor ratio 20:1). Squeeze it to leave in 80% on its weight of solution.
- 5.3 Dry the specimen by hanging it in an oven for 30 min. or longer if necessary, at  $60 \pm 2^{\circ}$ C. (140  $\pm$  4°F.). Then bake it by heating for 15 min. at  $105 \pm 2^{\circ}$ C. (221  $\pm$  4°F.).
- 5.4 Rinse the specimen for 5 min. in cold running tap-water and then divide it into two equal parts. Dry one half in air at a temperature not exceeding 60°c. (140°F.).
- 5.5 Agitate the other half for 30 min. at room temperature in the sodium carbonate solution (liquor ratio 40:1). Then rinse it for 5 min. in cold running water and dry it in air at a temperature not exceeding 60°C. (140°F.).
- 5.6 Assess the effect on the test-control specimen with the grey scale. If the change in colour is not equal to 2 yellower, the test has not been carried out correctly, and the operations described in §§5.1-5.7 inclusive must be repeated with fresh composite specimens and a fresh composite test-control specimen.
- 5.7 Assess the change in colour of each half of the specimen with the grey scale (see §7.2).

#### 6. Report

6.1 Report the numerical rating for change in colour for both the rinsed and the neutralised portions of the specimen.

#### 7. Notes

7.1 Test Control—A well wetted-out pattern of wool cloth is entered at 40°c. into a dyebath containing 1% Alizarin Red W powder, 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>42</sub>10H<sub>2</sub>O), and 3% acetic acid (30%), all percentages being calculated on the weight of the pattern, at a liquor ratio of 40:1.

The dyebath is raised to the boil in 30 min. and boiled for a further 30 min. If necessary, the dyebath is exhausted by careful addition of 1-3% acetic acid (30%) or 1% sulphuric acid (sp.gr. 1.84) well diluted with water. The bath is boiled for a further 15 min. after addition of the acid.

The dyebath is cooled down by addition of cold water, and 0.5% potassium dichromate dissolved in water is added. The dyebath is raised to the boil and boiled for 30 min.

The pattern is then removed, rinsed, and dried.

- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Chlorination, Acid

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Chlorination in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the manufacturing operation in which an acid hypochlorite solution is used to prevent wool textiles from shrinking.

#### 2. Principle

2.1 A specimen of the textile in contact with undyed textiles of interest is treated successively with solutions of hydrochloric acid, sodium hypochlorite, and sodium sulphite, rinsed, and dried. The change in colour of the specimen and the staining of the undyed textiles are assessed with standard grey scales. A test-control fabric is used.

#### 3. Apparatus and Reagents

3.1 Yarns of scoured unbleached undyed wool, undyed bleached cotton, and other fibres as desired for assessment of staining if fabrics or yarns are to be tested; comparable undyed fabrics if fibres are to be tested

- 3.2 Hydrochloric acid solution containing 6 ml. hydrochloric acid (sp.gr. 1-16) per litre
- 3.3 Sodium hypochlorite solution containing 1 g. of available chlorine per litre. Sodium hypochlorite of the following composition shall be used—

Sodium hypochlorite	NaOCI	14-16%
Sodium chloride	NaCl	12-17%
Sodium hydroxide	NaOH	2.0% maximum
Sodium carbonate	Na <sub>s</sub> CO <sub>s</sub>	2.0% maximum
Iron .	Fe	0-001 % maximum

- 3.4 Sodium sulphite solution containing 3 g. Na<sub>2</sub>SO<sub>3</sub>,7H<sub>2</sub>O per litre
- 3.5 Test Control—A dyeing of 1.5% Coomassie Blue RLS (C.I. 208) on wool cloth (see §7.1)
- 3.6 Grey scale for assessing change in colour and staining of undyed cloths (see §7.2).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, sew stitches of the undyed yarns (see §3.1) at intervals of approx. 1 cm. into a specimen of the fabric at least 7.5 cm. × 5 cm.
- 4.2 If the textile to be tested is yarn, knit it into fabric and make a composite specimen from it as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet at least 7.5 cm. × 5 cm. in size, place the sheet between the undyed wool and the undyed cotton or other fabrics, and sew the three together with stitching at intervals of 1 cm. The weight of the coloured textile should approximate to that of the undyed wool.
- 4.4 Test Control—Prepare a composite specimen of the test-control dyeing (see §3.5) in the way outlined for fabric in §4.1.

#### 5. Procedure

- 5.1 Carry out the operations described in §§5.2-5.5 inclusive with the composite specimens and the composite test-control specimen in parallel in separate baths.
- 5.2 Immerse the composite specimen in the hydrochloric acid solution (see §3.2) at a liquor ratio of 25:1 for 10 min. at room temperature.
- 5.3 Add an equal volume of the sodium hypochlorite solution (see §3.3) and keep the composite specimen immersed for a further 10 min.
- 5.4 Rinse the composite specimen thoroughly in cold running tap-water, and then immerse it in the sodium sulphite solution (see §3.4) for 10 min. at 35-40°C. (95-104°F.), at a liquor ratio of 50: 1.
- 5.5 Thoroughly rinse the composite specimen from §5.4 in cold running water and dry it in air at a temperature not exceeding 60°c. (140°F.).
- 5.6 Assess the effect on the test-control specimen with the grey scale. If the change in colour is not equal to 3, the test has not been carried out correctly, and the operations described in §§5.1-5.7 must be repeated with fresh composite specimens and a fresh composite test-control specimen.
- 5.7 Assess the change in colour of the specimen and the staining of the undyed materials with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical rating for change in colour and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 Test Control— A well wetted-out pattern of wool cloth is entered at 40°C. (104°F.) into a dyebath containing 1.5% Coomassie Blue RLS, 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>, 10H<sub>2</sub>O), and 3% acetic acid (30%), all percentages being calculated on the weight of the pattern, at a liquor ratio of 40:1.
  - The dyebath is raised to the boil in 30 min., and boiled for a further 30 min. If necessary, the dyebath is exhausted by careful addition of 1-3% acetic acid (30%) or 1% sulphuric acid

(sp. gr. 1.84) well diluted with water. The bath is boiled for a further 15 min, after addition of the acid.

The pattern is then removed, rinsed, and dried.

- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Light: Daylight

(PROPOSED ISO RECOMMENDATION)

Note—This test is identical with B.S. 1006:1955. An account of the development of the light fastness standards is given in J.S.D.C., 67, 188 (1951)

#### 1. Purpose and Scope

- 1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds and in all forms to the action of daylight (see §7.1).
- 1.2 The method may be used for rating dyes by applying the dye to textiles under specified conditions and at a specified depth of colour and testing the dyed textiles.

#### 2. Principle

2.1 A specimen of the textile is exposed to daylight under prescribed conditions, including protection from rain, along with eight dyed wool standards. The fastness is assessed by comparing the fading of the textile with that of the standards.

#### 3. Standards and Equipment

- 3.1 Standards-
- 3.1.1 The standards for this test are blue wool cloths dyed with the dyes listed below. They range from No. 1 (very low light fastness) to No. 8 (very high light fastness) (see §7.2).

Fastness Rating	Dye
1	Acilan Brilliant Blue FFR (FBy)
2	Acilan Brilliant Blue FFB (FBy)
3	Coomassie Brilliant Blue R (ICI)
4	Supramin Blue EG (FBy)
5	Solway Blue RN (ICI)
6	Alizarine Light Blue 4GL (S)
7	Soledon Blue 4BC powder (ICI)
8	Indigosol Blue AGG (DH)

- 3.1.2 Master sets of the standard dyeings are to be filed in each country for reference.
- 3.2 Exposure rack, facing due south in the Northern Hemisphere, north in the Southern Hemisphere, and sloping at an angle of 45°, in a location such that shadows of surrounding objects will not fall on the exposed textiles, covered with window glass to protect the specimens from the weather, the glass being not less than 2.5 cm. from the specimen and provided with adequate ventilation.
- 3.3 Opaque cardboard or other thin opaque material, e.g. thin sheet aluminium, or cardboard covered with aluminium foil.
- 3.4 Grey Scale for assessing Change in Colour (see §7.3).

#### 4. Specimen

- 4.1 An area of the textile not less than 1 cm. × 6 cm. is used when the several periods of exposure are made side by side on the same piece, which is the preferred practice. The specimen may be—a strip of cloth, yarns wound close together on a card or laid parallel and fastened on a card, or a mat of fibres combed and compressed to give a uniform surface and fastened on a card.
- 4.2 To facilitate handling, the specimen or specimens to be tested and similar strips of the standards may be mounted on a card in some such arrangement as indicated in §7.5.

#### 5. Procedure

- 5.1 Expose the specimen (or a group of specimens) and the standards simultaneously to daylight under the conditions enumerated in §3.2 in such a manner and for such times as are necessary to evaluate fully the light fastness of each specimen relative to that of the standards, by successively covering the specimens and exposed standards throughout the time of the test. (See §7.5 for two suggested methods of procedure.)
- 5.2 Preliminary Assessment of Light Fastness—When a change in colour (see §7.6) of the specimen under test can just be perceived, note the number of the standard showing a similar change; alternatively, when a change in colour of Standard 3 can just be perceived, assess any change that has occurred in the specimen in comparison with the changes in Standards 1, 2, and 3 (see §7.4). Continue to expose after the preliminary assessment as required to evaluate fully the light fastness.
- 5.3 Assessment of Light Fastness—Compare the changes in colour of the test specimen with the changes which have occurred in the standards (see §7.6). The light fastness of the specimen is the number of the standard which shows similar changes in colour (visual contrast between exposed and unexposed parts in the test). If the specimen shows changes in colour intermediate between two standards, an appropriate half-rating may be given; e.g. a light fastness of 3-4 means that the textile is not as fast as Standard 4, but is faster than Standard 3.
- 5.4 If the colour of the specimen is more fugitive than that of Standard No. 1, a rating of 1 is given.
- 5.5 If the light fastness is 4 or higher, the assessment based on the first perceptible change (see §5.2) becomes significant; if this preliminary assessment is 3 or lower, it is to be included in the rating in brackets. For example, a rating of 6 (3) indicates that the specimen changes very slightly in the test when Standard 3 just begins to fade, but that on continuing the exposure the resistance to light is equal to that of Standard 6.

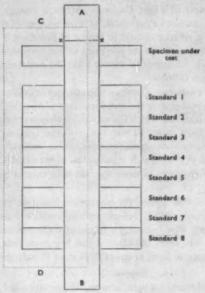
#### 6. Report

6.1 Report the numerical rating for light fastness. If this rating is 4 or higher and the preliminary assessment is 3 or lower, report the latter figure in brackets.

#### 7. Notes

- 7.1 The use of fading lamps rather than daylight is common practice, and the results obtained with such tests are undoubtedly useful where speed is essential. The results obtained for many textiles differ, however, from the results obtained with daylight exposure.
  - A low-pressure mercury-vapour lamp must not be used, as its emission spectrum is very different from that of daylight and leads to incorrect results.
- 7.2 Sets of the light-fastness standards can be obtained from the British Standards Institution, British Standards House, 2 Park Street, London W.1, or Coronation House, Market Street, Manchester 1. They are specially dyed to match a master set of standards in colour and in fading behaviour. It has been found that, when repeated dyeings of the dyed standards are made, the amount of dye required to match the previous lot is often different from that originally used. The dyeing strengths would therefore be misleading, and they are intentionally omitted from this listing.
- 7.3 See the following sections—
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.
- 7.4 Phototropic specimens must be allowed to condition in the dark at room temperature for 2 hr. before assessing the change in colour.
- 7.5 Suggested Procedures for Mounting and Testing for Light Fastness-
- 7.5.1 Method 1-
- 7.5.1.1 This method is considered ideal and should be used in cases of dispute. It requires one set of standards for each specimen under test and is therefore impracticable when a large number of specimens have to be tested concurrently; in such cases, Method 2 (see §7.5.2) is suggested.

7.5.1.2 Arrange the specimen to be tested and the standards as shown in Fig. 1 with an opaque cover across the middle third of the specimen and standards. Expose to daylight under the conditions enumerated in §3.2. Follow the effect of light by removing the cover and inspecting the specimen frequently. When a change can just be perceived, note the number of the standard showing a similar change.



- AB Opaque cover. May be hinged at xx so that it can be lifted and returned to the same place over the specimen and standards.
- CD Second cover.

Fig. 1- Mounting for Method 1

- 7.5.1.3 Continue to expose until the contrast between the exposed and the covered portion of the specimen is equal to the contrast between the two grey colour chips illustrating Grade 4 on the grey scale (see §7.3). Cover a second one-third of the specimen and standards with an additional opaque cover (CD in Fig. 1).
- 7.5.1.4 Continue to expose until the contrast between the fully exposed and the unexposed portion is equal to Grade 3 on the grey scale. If Standard 7 fades to a contrast equal to Grade 4 on the grey scale before the specimen does, the exposure may be concluded at this stage. (When a specimen has a light fastness of 7 or over, it would require unduly long exposure to produce a contrast equal to Grade 3; moreover, this contrast would be impossible to obtain when the light fastness is 8. Assessments in the region 7 to 8 are, therefore, made when the contrast produced on Standard 7 is equal to Grade 4, the time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.)
- 7.5.1.5 Assessment of Light Fastness— The specimen now shows three separate areas— an unexposed region and two areas which have been changed to different degrees. Compare these changes with the changes which have occurred in the standard, and rate the light fastness as in §5.3.
- 7.5.2 Method 2-
- 7.5.2.1 This method should be used when a large number of specimens have to be tested concurrently. It enables a number of specimens differing in light fastness to be rated against a single set of standards.

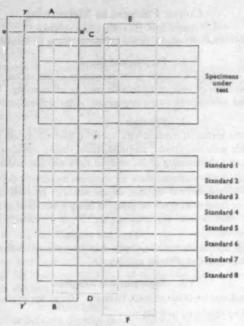


Fig. 2- Mounting for Method 2

- 7.5.2.2 Arrange the specimens to be tested and the standards as shown in Fig. 2, with card AB covering one-quarter of the total length of each specimen and standard. Expose to daylight under the conditions enumerated in §3.2. Follow the effect of light by lifting the cover periodically and inspecting the standards. When a change in Standard 3 can just be perceived, inspect the specimens and rate their light fastness by comparing any change that has occurred with the changes that have occurred in Standards 1, 2, and 3.
- 7.5.2.3 Replace the card AB in exactly the same position, and continue to expose until a change in Standard 4 can just be perceived; at this point fix an additional cover CD in the position shown in Fig. 2, overlapping the first cover.
- 7.5.2.4 Continue to expose until a change in Standard 6 can just be perceived; then fix the final cover EF in the position shown, the other two covers remaining in position. Expose until either-(a) a contrast is produced on Standard 7 equal to the contrast illustrated by Grade 4 of the grey scale, or (b) a contrast equal to Grade 3 has been produced on the most resistant specimen, whichever occurs first. Remove the three covers.
- 7.5.2.5 Compare the changes in the specimens with those in the standards, and rate the light fastness as in §5.3.
  - The term change in colour includes not only true "fading", i.e. destruction of dyes, but also changes in hue, depth, brightness, or any combination of these characteristics of colour. If the difference in colour is a change of hue or brightness, this can be indicated by adding abbreviations, as follows, to the numerical colour fastness rating-

$$BI = Bluer$$
  $R = Redder$   $D = Duller$   $G = Greener$   $Y = Yellower$   $Br = Brighter$ 

#### Colour Fastness to Mercerising

(Proposed ISO RECOMMENDATION)

Note- This test replaces the test for Mercerising in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of yarns to the action of concentrated solutions of sodium hydroxide used in mercerising. It is mainly applicable to cotton yarns and mixed-fibre yarns containing other natural cellulose fibres with cotton.

#### 2. Principle

- 2.1 A specimen of the textile in contact with specified undyed cotton cloth is treated with the sodium hydroxide solution, rinsed, acidified, again rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloth are assessed with standard grey scales.
- 2.2 As completely resistant specimens show an apparent increase in depth of colour, these would not be rated 5 by the normal method of assessment. In such cases, therefore, only the changes in hue or brightness shall be assessed using a standard grey scale, and such assessments shall be marked with an asterisk, e.g. 5\*, 3-4 redder\*, or 2 bluer, duller\*, and explained in a footnote.
- 2.3 Specimens which do not increase in depth shall be assessed in the normal manner and the results shall not be marked with an asterisk.

#### 3. Apparatus and Reagents

- 3.1 Undyed, bleached cotton cloth at least 10 cm. × 10 cm. for evaluating staining
- 3.2 Frame for holding specimen (see §4)
- 3.3 Sodium hydroxide solution, 300 g. sodium hydroxide per litre
- 3.4 Sulphuric acid solution, 5 ml. concentrated sulphuric acid (sp. gr. 1-84) per litre
- 3.5 Grey scales for assessing change in colour and staining of undyed cloths (see §7.1).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, sew a specimen of it at least 10 cm. × 10 cm. to an equalsized piece of the undyed cloth (§3.1) along all four sides. Fasten this composite specimen to a frame firmly, but without excessive tension, with the coloured material uppermost.
- 4.2 If the textile to be tested is yarn or thread, wind it on a rigid frame firmly, but without excessive tension, with the strands close together and parallel to provide an area at least 10 cm. × 10 cm. Sew an equal-sized piece of the undyed cloth to this area along the two sides across the strands, with the coloured material uppermost.

#### 5. Procedure

- 5.1 Immerse the composite specimen in the sodium hydroxide solution at 20  $\pm$  2°c. (68  $\pm$  4°F.) for 5 min.
  - Rinse it by pouring on 1 litre of water at  $70 \pm 2^{\circ}$ c. (158  $\pm 4^{\circ}$ F.) over a period of 1 min., and then rinsing in cold running tap-water for 5 min.
- 5.2 Remove it from the frame and immerse it in the sulphuric acid solution for 5 min. at a liquor ratio of 50:1. Wash it in cold running water until neutral.
- 5.3 Remove the stitching along three sides (one side for yarns and threads), and dry in air at a temperature not exceeding 60°C. (140°F.), taking care that the undyed cloth and the coloured material are kept apart except at the stitching.
- 5.4 If the specimen shows increased depth of colour, assess the change in hue or brightness only, using the appropriate grey scale. Assess the staining of the undyed cotton cloth with the appropriate grey scale (see §7.1).
- 5.5 If the specimen does not show increased depth of colour, assess the change as an overall contrast and the staining of the undyed cotton cloth with the grey scales (see §7.1).

#### 6. Report

- 6.1 In the case of assessments in accordance with §5.4, report and mark with an asterisk any change in hue or brightness of the specimen, and report the numerical rating for staining of the undyed cotton cloth.
- 6.2 In the case of assessments in accordance with §5.5, report the numerical rating for change in colour and the numerical rating for staining of the undyed cotton cloth.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Metals in the Dyebath: Chromium Salts

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Chrome (Dichromate) in Dyebath in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the effect on the colour of a dye of dyeing in the presence of sexivalent chromium salts. Alternative conditions are specified in §5.3 to provide a milder test suitable for assessing the effect of chromium salts in such concentrations as might be found when shading.

#### 2. Principle

2.1 The difference in colour between dyeings made with and without potassium dichromate is assessed with the standard grey scale.

#### 3. Apparatus and Reagents

- 3.1 Two pieces of undyed wool cloth of a size suitable for laboratory dyeing
- 3.2 Two dyebaths and solutions usual for the dye
- 3.3 Potassium dichromate solution containing 1% K<sub>2</sub>Cr<sub>2</sub>O<sub>2</sub>
- 3.4 Grey Scale for assessing Change in Colour (see §7.1).

#### 4. Specimen

4.1 See §3.1.

#### 5. Procedure

- 5.1 Make two dyeings of the dye at the standard depth of colour on the wool cloth using the normal method for the dye under examination and a liquor ratio of 40:1.
- 5.2 Method 1—On completion of the dyeing add to one of the dyebaths sufficient potassium dichromate solution to give 1% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> on the weight of the wool. Maintain both baths at the boil for a further 60 min.
- 5.3 Method 2— On completion of the dyeing add to one of the dyebaths sufficient potassium dichromate solution to give 0.2% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> on the weight of the wool. Maintain both baths at the boil for a further 60 min.
- 5.4 Compare the colour of the dyeing made in the presence of potassium dichromate with that of the dyeing made in its absence, and assess the difference with the grey scale.

#### 6. Report

6.1 Report the dye used, the method and the strength of dyeing, and the numerical rating of the change in colour.

#### 7. Note

- 7.1 See the following sections—
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Metals in the Dyebath: Iron and Copper

(PROPOSED ISO RECOMMENDATION)

NOTE- This test replaces the test for Metals (Iron and Copper) in Dyebath in the Second Report

1. Purpose and Scope

1.1 This method is intended for assessing the effect on the colour of a dye of dyeing in the presence of metals either used in the construction of dyeing machinery or resulting from water and steam used in dyeing.

2. Principle

2.1 The difference in colour between dyeings made in the presence and in the absence of salts of the metal is assessed with the standard grey scale.

3. Apparatus and Reagents

- 3.1 Three pieces of undyed light wool cloth of a size suitable for laboratory dyeing
- 3.2 Three dyebaths and solutions usual for the dye
- 3.3 Copper sulphate solution containing 0.2% CuSO<sub>4</sub>,5H<sub>2</sub>O on the weight of one piece of the wool (see §3.1)
- 3.4 Ammonium ferric sulphate solution containing 0.5% (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>,Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>,24H<sub>2</sub>O on the weight of one piece of the wool (see §3.1)
- 3.5 Grey Scale for assessing Change in Colour (see §7.1).

#### 4. Specimen

4.1 See §3.1.

#### 5. Procedure

- 5.1 Make three dyeings at the standard depth of colour on the wool cloth according to the usual method for the dye under examination. Before entering the fabric, add the ammonium ferric sulphate to one dyebath and the copper sulphate to another, and bring the liquor ratio in each bath to 40:1.
- 5.2 Compare the colours of the dyeings made in the presence of copper sulphate and of ferric sulphate with that of the dyeing made in their absence, and assess the differences with the grey scale.

6. Report

6.1 Report the dye used, the method and the strength of dyeing, and the numerical rating of the change in colour.

#### 7. Note

7.1 See the following sections-

- (a) Colour Fastness of Textiles: General Principles of Testing
- (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Organic Solvents

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Organic Solvents in the Second Report

1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to organic solvents, and in particular to solvents used in cleaning or mill processing.

2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in the solvent. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

3. Apparatus and Reagents

- 3.1 Suitable container with means of agitation (see §7.1)
- 3.2 Specified solvent (see §7.2)

3.3 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified—

If First Piece is-	Second Piece to he-	If First Piece is-	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.4 Grey scales for assessing change in colour and staining of undyed cloths (see §7.3).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of cloth required in §3.3 and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm.
  × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Agitate the composite specimen continuously for 30 min. in the solvent at room temperature with a liquor ratio of 40:1. If the agitation is by hand, the specimen must be pressed against the container every 2 min. with a glass rod (see §7.1) without removing the specimen from the solvent.
- 5.2 Squeeze excess solvent from the specimen and dry it in air at 80 ± 2°c. (176 ± 4°F.) without unstitching. Take the precautions necessary for safety in drying inflammable or explosive solvents.
- 5.3 Remove the stitching, and assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §3.4).

#### 6. Report

6.1 Report the solvent used, the numerical rating for change in colour, and the numerical rating for colour staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 A 500-ml. beaker or other suitable open container may be used for the test, agitation being by hand with a glass rod flattened at one end. A closed vessel agitated by shaking or tumbling in a hand- or motor-driven machine may be used.
- 7.2 It is anticipated that the test will be made in European countries with trichloroethylene and in the U.S.A. with tetrachloroethylene (perchlorethylene) and with Stoddart solvent (specially specified naphtha for the use of dry cleaners). Other solvents for which information is desired may be used. Trichloroethylene should be used for rating dyes. Halogenated hydrocarbons should be stored over anhydrous sodium carbonate to neutralise hydrochloric acid.
- 7.3 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### **Colour Fastness to Potting**

(PROPOSED ISO RECOMMENDATION)

Nors- This test replaces the test for Potting in the Second Report)

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of wool and textiles containing wool to the action of boiling water.

#### 2. Principle

2.1 A specimen of the textile between undyed wool and cotton cloths is rolled around a glass rod and treated with boiling water. The cloths are separated and dried, and the change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Vessel equipped with reflux condenser to hold a cylindrical specimen 4 cm. long in boiling water
- 3.2 Glass rod 5-8 mm, in diameter
- 3.3 Undyed unbleached wool cloth 10 cm. × 4 cm.
- 3.4 Undyed bleached cotton cloth or the fibre used in admixture with the wool
- 3.5 Grey scales for assessing change in colour and staining of undved cloths (see §7.1).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of undyed cloth required in §§3.3 and 3.4, and sew along one of the shorter sides.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Roll the composite specimen around the glass rod to form a cylinder 4 cm. long and tie it loosely with thread.
- 5.2 Treat the specimen on the rod for 1 hr. in boiling distilled water, at a liquor ratio of 30:1, under reflux.
- 5.3 Remove the specimen from the rod and separate the undyed cloths from it. Dry all three in air at a temperature not exceeding 60°C. (140°F.).
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.1).

#### 6. Report

6.1 Report the numerical rating for change in colour and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Pressing: Hot Pressing

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the tests for Hot Pressing in the Second Report

#### 1. Purpose and Scope

- 1.1 This method is intended for assessing the resistance of the colour of textile yarns, thread, and fabrics of all kinds to ironing and to processing on hot cylinders.
- 1.2 Tests are given for hot pressing when the textile is wet, when it is damp, and when it is dry.

#### 2. Principle

2.1 For dry pressing, the dry specimen on a dry undyed cotton cloth is pressed with an iron of specified weight and temperature. For wet pressing, the wet specimen placed on a dry undyed cotton cloth and covered with a wet undyed cotton cloth is pressed with the iron. For damp

pressing, the dry specimen is placed on a dry undyed cotton cloth, covered with a wet undyed cotton cloth, and pressed with the iron. The change in colour and the staining of the undyed cloths are assessed with standard grey scales immediately and again after a period of exposure to air.

#### 3. Apparatus and Reagents

3.1 Hand iron of the proper weight to give a pressure of approx. 30 g./sq.cm. (0-43 lb./sq. in.) (see §7.1) at the temperature indicated in the following table (see §7.2)—

Fabric	Temperature	
	(°c.)	(°F.)
Cotton and linen	190-210	374-410
Wool, silk, and viscose rayon	140-160	284-320
Acetate rayon and polyamide fibres	115-120	239-248

- 3.2 Undyed bleached cotton cloth weighing approx. 125 g./sq.metre (3.7 oz./sq.yd.). Five pieces not less than 14 cm. × 4 cm. are required
- 3.3 Grey scales for assessing change in colour and staining (see §7.3).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a piece 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn or thread, knit it into fabric and use a piece 10 cm. × 4 cm., or wind it around a piece of thin cardboard 14 cm. × 4 cm. to obtain the area of the textile for test.

#### 5. Procedure

- 5.1 Specimens of materials that have been subjected to any heat or drying treatment must be allowed to condition (by exposure to air at 65% relative humidity and 20  $\pm$  2°c.) before they are tested.
- 5.2 Dry Pressing—Place the specimen on the piece of dry cotton cloth (see §3.2) on a smooth horizontal surface. Place the iron on top and leave it for 15 sec.
- 5.3 Wet Pressing— Soak the specimen and a piece of the cotton cloth (§3.2) in distilled water, and squeeze or extract them to contain their own weights of water. Place the wet specimen on a piece of the dry cotton cloth (see §3.2) and place the wet cotton cloth on the specimen. Press by moving the iron to and fro over the wet cloth (without additional pressure) for 15 sec.
- 5.4 Damp Pressing—Place the dry specimen on a piece of the dry cotton cloth (see §3.2). Soak another piece of the undyed cloth in distilled water, and squeeze or extract it to contain its own weight of water. Place the wet cloth on the specimen. Press by moving the iron to and fro over the wet cloth (without additional pressure) for 15 sec.
- 5.5 Assess the change in colour of the specimen with the appropriate grey scale (see §7.3) immediately and again after the specimen has been allowed to condition for 4 hr. in the standard atmosphere, viz. 65% relative humidity and  $20 \pm 2^{\circ}$ c. (68  $\pm 4^{\circ}$ F.).
- 5.6 Assess the staining of the undyed cloths with the appropriate grey scale (see §7.3). In wet pressing (see §5.3) and in damp pressing (see §5.4) the staining shall be assessed on the undyed cloth which shows the greater amount of staining.

#### 6. Report

6.1 Report the numerical rating for the change in colour for dry, wet, and damp pressing immediately after testing and after conditioning for 4 hr. Report the numerical rating of the staining of the undyed cloth.

#### 7. Notes

- 7.1 The pressure on the cloth under test will depend upon the area of the bottom of the iron lying over the 14 cm. × 4 cm. specimen.
- 7.2 The measurement of the surface temperature of the iron shall be made by means of an electric surface pyrometer or by observing the behaviour of crystals such as m-hydroxybenzoic acid (m.p. 200°c., 392°F.), citric acid (m.p. 153°c., 307°F.), or benzoic acid (m.p. 122°c., 252°F.) when placed on the iron.

- 7.3 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Rubbing

(PROPOSED ISO RECOMMENDATION)

NOTE- This test replaces the test for Rubbing in the Second Report

#### 1. Purpose and Scope

- 1.1 This method is intended for assessing the resistance of the colour of textiles in all forms to rubbing off and staining other materials.
- 1.2 Two tests are made, one with a dry rubbing cloth and one with a wet rubbing cloth.

#### 2. Principle

2.1 Specimens of the textile are rubbed with dry undyed cotton cloth and with wet undyed cotton cloth. The staining of the undyed cloths is assessed with the standard grey scale.

#### 3. Apparatus and Reagents

- 3.1 Crockmeter or its equivalent (see §7.1)
- 3.2 Undyed bleached cotton lawn or similar cloth free from starch or other finish, cut into 5-cm. (2-in.) squares (see §7.2).
- 3.3 Grey Scale for assessing Staining of undyed cloth (see §7.3).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, two pieces not less than 14 cm. × 5 cm. are required for dry rubbing and two for wet rubbing. One specimen of each pair has the long direction parallel to the warp yarns, the other parallel to the west yarns.
- 4.2 If the textile to be tested is yarn or thread, knit it into fabric of suitable dimensions (see §4.1), or form a layer of parallel strands by wrapping it lengthwise on a cardboard rectangle of suitable dimensions.
- 4.3 If the textile to be tested is in the form of loose fibre, comb some of it into a mat of fibre of suitable dimensions (§4.1) at least 1 cm. thick in the uncompressed state. The mat should then be secured to a rectangle of cardboard.

#### 5. Procedure

- 5.1 Dry Rubbing— With the dry undyed cloth in place over the end of the finger of the testing device, rub it to and fro in a straight line along a track 10 cm. (4 in.) long on the dry specimen, 10 times in 10 sec., with a downward force on the finger of 900 g. (2 lb.). Test warp and weft directions separately.
- 5.2 Wet Rubbing— Repeat the test with a fresh dry specimen and undyed cloth which has been wetted with water and squeezed to a take-up of 100%. Dry the cloths at room temperature.
- 5.3 Assess the staining of the undyed cloths with the grey scale (see §7.3).

#### 6. Report

6.1 Report the numerical rating for dry staining and for wet staining for the direction, warp or weft, showing heavier staining.

#### 7. Notes

- 7.1 The Crockmeter may be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 Cloth for the purpose may be obtained from The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.
- 7.3 See the following sections-
  - (a) Colour Fastness of Textiles; General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Soda Boiling

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the test for Soda Boiling given in the Second Report

#### 1. Purpose and Scope

- 1.1 This method is intended for assessing the resistance of the colour of textiles in all forms to the action of boiling dilute sodium carbonate solutions. It is mainly applicable to natural and regenerated cellulose materials.
- 1.2 Two tests are given, one with and the other without the addition of a reduction inhibitor.

#### 2. Principle

2.1 A specimen of the textile between specified undyed cloths is rolled around a glass rod and treated with boiling sodium carbonate solution with and without the addition of a reduction inhibitor. The composite specimen is rinsed, the cloths are separated and dried, and the change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Vessel equipped with water-cooled reflux condenser of the finger type to hold a cylindrical specimen 4 cm. long in the boiling solution
- 3.2 Glass rod 5-8 mm, in diameter
- 3.3 Desized undyed cotton fabric
- 3.4 Undyed cloth of the type under test (or, if fibre or yarn is being tested, undyed cloth made from the same kind of fibre)
- 3.5 Sodium carbonate solution containing 10 g. anhydrous sodium carbonate per litre
- 3.6 Sodium carbonate solution containing 10 g. anhydrous sodium carbonate per litre and 4 g. sodium m-nitrobenzenesulphonate per litre
- Test-control Specimens Dyeings of Solanthrene Brilliant Pink R Powder (C.I. 1211) (see §7.1)
- 3.8 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

#### 4. Specimen

- 4.1 Two specimens, prepared as follows, are required.
- 4.2 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between pieces of the undyed cloths specified in §§3.3 and 3.4, and sew along one of the shorter sides.
- 4.3 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.2, or form a layer of parallel lengths of it between the two undyed cloths and sew along one of the shorter sides to hold the yarns in place.
- 4.4 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew along one of the shorter sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Carry out the operation described in §§5.2-5.4 inclusive with each specimen and the test-control specimen in parallel in separate baths.
- 5.2 Roll the composite specimen around the glass rod to form a cylinder 4 cm. long and tie it loosely with thread.
- 5.3 Treat one composite specimen on the rod by boiling gently under reflux for 1 hr. in the sodium carbonate solution (§3.5) at a liquor ratio of 30:1. Treat the other composite specimen in the same way and for the same time in boiling sodium carbonate solution containing the sodium m-nitrobenzenesulphonate (§3.6).
- 5.4 Remove the specimens from the rod immediately, rinse for 10 min. in cold running tap-water, and separate the undyed cloths from them. Dry all pieces in air at a temperature not exceeding 60°c, (140°F.).

- 5.5 Assess the effect on the test-control specimens with grey scales. The ratings of a control specimen after boiling with sodium m-nitrobenzenesulphonate should be 3-4 weaker, yellower in respect of change in colour and 5 in respect of staining. The ratings of a control specimen after boiling without sodium m-nitrobenzenesulphonate should be 2-3 weaker, yellower in respect of change in colour and 2-3 in respect of staining. If the control specimens do not yield these values, the test has not been carried out correctly, and the operations described in §§5.1-5.4 inclusive must be repeated with fresh composite specimens and fresh composite test-control specimens.
- 5.6 Assess the changes in colour of the specimens and the staining of the undyed cloths with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical ratings for change in colour and the numerical ratings for staining of each kind of undyed cloth tested with sodium carbonate alone and with sodium carbonate and sodium m-nitrobenzenesulphonate reduction inhibitor.

#### 7. Notes

#### 7.1 Test Control-

Reduction—3.0% Solanthrene Brilliant Pink R Powder is pasted with 150 times its weight of water using an anionic wetting agent at the rate of 3 ml. per gram of dye. 40 ml. of caustic soda (76° Tw., 36° Bé.) and 13 g. of sodium dithionite (sodium hydrosulphite) are added per litre, and the dye is allowed to reduce for 15 min. at 50°c.

**Dyeing**— The dyebath is set at a liquor ratio of 25:1. To it are added 2-3 ml. of sodium hydroxide (76° Tw., 36° Bé.) and 1 g. of sodium dithionite (hydrosulphite) per litre, followed by the calculated amount of reduced dye. The dyeing is started at 30°c., and heat is applied for 15 min. to bring the temperature to 60°c. Dyeing is continued at this temperature for 30 min.

The pattern is then oxidised in air, rinsed in cold water, soaped at the boil, and dried.

#### 7.2 See the following sections-

- (a) Colour Fastness of Textiles: General Principles of Testing
- (b) Colour Fastness: Grey Scale for assessing Change in Colour
- (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Stoving

(PROPOSED ISO RECOMMENDATION)

NOTE— This test replaces the test for Stoving in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of wool, silk, and mixed-fibre textiles containing one of these fibres to the action of sulphur dioxide as used for bleaching animal fibres.

#### 2. Principle

2.1 A specimen of the textile containing its own weight of soap solution and a control are exposed in an atmosphere containing sulphur dioxide. The change in colour of the specimen is assessed with a standard grey scale.

#### 3. Apparatus and Reagent

- 3.1 Vessel of approx. 10 litres capacity for the sulphur dioxide atmosphere
- 3.2 Sulphur
- 3.3 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight—

Free alkali calculated as Na<sub>2</sub>CO<sub>3</sub>
Free alkali calculated as NaOH
Combined fatty acids calculated as Na salts
Titre of mixed fatty acids prepared from the soap
Iodine value

0.3% maximum 0-1% maximum 85% minimum

30°C. maximum 50 maximum

- 3.4 Soap solution, containing 5 g. of soap (see §3.3) per litre of distilled water
- 3.5 Test Control—A dyeing on wool of 2.5% Coomassie Milling Scarlet 5 BS (C.I. 487) (see §7.1)
- 3.6 Grey Scale for assessing Change in Colour (see §7.2).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a specimen at least 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a specimen at least 10 cm. × 4 cm., or from a number of lengths of it form a wick 10 cm. long and about 0.5 cm. in diameter tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

#### 5. Procedure

- 5.1 Thoroughly impregnate the specimen and the test-control specimen by immersion for 5 min. at  $25 \pm 2^{\circ}c$ . (77  $\pm 4^{\circ}F$ .) in the soap solution. Then squeeze so that each contains its own weight of solution.
- 5.2 Suspend the specimen and the test control (see §3.5) for 16 hr. in the vessel containing an atmosphere of sulphur dioxide, obtained by igniting 5 g. of sulphur below the specimen and the control and immediately closing the vessel.
- 5.3 Remove the specimen and the control from the sulphur dioxide atmosphere, and allow to hang in air for at least 2 hr. without rinsing. The specimen and the control are to be examined when dry. If the specimen contains cellulosic fibres, rinse it in cold running tap-water and dry in air at a temperature not exceeding 60°c. (140°F.).
- 5.4 Assess the effect on the test control with the grey scale. If the change is not equal to the contrast illustrated by Grade 3 on the grey scale, the test has not been carried out correctly, and the operations decribed in §§5.1-5.5 inclusive must be repeated with fresh composite specimens and a fresh composite test-control specimen.
- 5.5 Assess the change in colour of the specimen with the grey scale (see §7.2).

#### 6. Report

6.1 Report the numerical rating for the colour change in the test.

#### 7. Notes

- 7.1 Test Control—A well wetted-out pattern of wool cloth is entered at 40°C. (104°F.) into a dyebath containing 2.5% Coomassie Milling Scarlet 5BS (C.I. 487), 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O), and 3% acetic acid (30%), all percentages being calculated on the weight of the pattern. The liquor ratio is 40:1. The dyebath is raised to the boil in 30 min. and boiled for 30 min. If necessary, the dyebath is exhausted by careful addition of 1-3% acetic acid (30%) or 1% sulphuric acid (sp. gr. 1.84) well diluted with water. The bath is boiled for a further 15 min. after addition of the acid. The pattern is then removed, rinsed, and dried.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### Colour Fastness to Washing: Hand Washing

(PROPOSED ISO RECOMMENDATION)

Note—This test replaces Washing Test No. I for Natural and Regenerated Cellulose and Washing Test No. I for Wool in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the action of soap solutions as used in washing them by hand.

#### 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a soap solution, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Suitable container with means of agitation (see §7.1)
- 3.2 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na<sub>2</sub>CO<sub>a</sub>

Free alkali calculated as NaOH

Combined fatty acids calculated as Na salts

Titre of mixed fatty acids prepared from the soap

30°c. maximum

\*Soap complying with B.S. 1912: 1953 is suitable for use in this test.

- 3.3 Soap solution, containing 5 g. of soap (see §3.2) per litre of distilled water
- 3.4 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is—	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.5 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of undyed cloth required in §3.4, and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Agitate the composite specimen continuously for 30 min. in the soap solution at 40 ± 2°C. (104 ± 4°F.) at a liquor ratio of 50: 1. If the agitation is by hand, press the specimen every 2 min. against the container without removing it from the solution. If mechanical agitation is used (see §7.1), the time of treatment, and if necessary the liquor ratio, shall be adjusted to give results identical with those of the hand test.
- 5.2 Rinse the composite specimen twice in cold distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not above 60°C. (140°F.), with the three parts in contact only along the one row of stitching.
- 5.3 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 A 500-ml. beaker or other suitable open container may be used for the test, agitation being by hand with a glass rod flattened at one end. A closed vessel agitated by shaking or tumbling by hand or a motor-driven machine may be used.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Water

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the tests for Water in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to immersion in water.

#### 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is immersed in water, drained, and placed in a Perspirometer or equivalent apparatus (see §7.1). The specimen and the undyed cloths are dried separately. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Perspirometer or equivalent apparatus (see §7.1)
- 3.2 Two undyed cloths each 6.5 cm. × 6.5 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is—	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
I inen	Wool	Polyester fibre	Wool

- 3.3 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2)
- 3.4 Oven maintained at 37  $\pm$  2°c. (100  $\pm$  4°F.).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 6.5 cm. x 6.5 cm. between two pieces of undyed cloth (see §3.2), and sew along one side to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew along two opposite sides near the ends of the yarn to form a composite specimen.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 6.5 cm. × 6.5 cm. in size, place the sheet between the two undyed cloths, and sew along two opposite sides to form a composite specimen.

#### 5. Procedure

- 5.1 Thoroughly wet the composite specimen in distilled water at room temperature. Pour off the water, and place the composite specimen between two glass plates measuring about 7.5 cm. × 6.5 cm. under a force of about 4.5 kg. (10 lb.).
- 5.2 Place the apparatus containing the composite specimen in the oven for 4 hr. at  $37 \pm 2^{\circ}$ C. (100  $\pm 4^{\circ}$ F.), or leave at room temperature for sufficient time to give equivalent results.
- 5.3 Separate the specimen and the undyed cloth, and dry them apart in air at a temperature not exceeding 60°C. (140°F.).
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloth with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical rating for colour change and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 The Perspirometer, developed by the American Association of Textile Chemists and Colorists, is intended for carrying out tests of this kind, but any apparatus giving identical results may be used. The "plate method" in the Second Report gives identical results. If it is to be used, §§5.1 and 5.2 should be replaced by the following—
  - 5.1 Thoroughly wet the composite specimen in distilled water at room temperature. Lay it out smooth in a flat-bottomed dish (area of bottom greater than 6.5 cm. × 6.5 cm.), and cover it with distilled water. Place a smooth glass plate (5 cm. × 5 cm. or slightly larger weighing approx. 50 g.) on it, and press evenly and lightly with the fingers to remove air bubbles. Allow to stand for 15 min. at room temperature.
  - 5.2 Pour off the water without removing the glass plate. Allow the specimen to remain under the plate for 4 hr. at  $37 \pm 2^{\circ}$ C.  $(100 \pm 4^{\circ}$ F.).
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Sea Water

(PROPOSED ISO RECOMMENDATION)

Note- This test replaces the tests for Sea Water in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to immersion in sea water.

#### 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is immersed in a solution containing sodium chloride, drained, and placed in a Perspirometer or equivalent apparatus (see §7.1). The specimen and the undyed cloths are dried separately. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Perspirometer or equivalent apparatus (see §7.1)
- 3.2 Two undyed cloths each 6.5 cm. × 6.5 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below or as otherwise specified (see §7.2)—

If First Piece is-	Second Piece to be-	If First Piece is-	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Dolumeter fibre	Wool

- 3.3 Solution of distilled water containing 30 g. of sodium chloride per litre
- 3.4 Standard grey scales (see §7.2)
- 3.5 Oven maintained at 37  $\pm$  °2°C. (100  $\pm$  4°F.).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 6.5 cm. × 6.5 cm. between two pieces of undyed cloth (see §3.2) and sew along one side to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew along two opposite sides near the ends of the yarn to form a composite specimen.

4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 6.5 cm. × 6.5 cm. in size, place the sheet between the two undyed cloths, and sew along two opposite sides to form a composite specimen.

#### 5. Procedure

- 5.1 Thoroughly wet the composite specimen in the solution (see §3.3) at room temperature. Pour off the solution, and place the composite specimen between two glass plates measuring about 7.5 cm. × 6.5 cm. under a force of about 4.5 kg. (10 lb.).
- 5.2 Place the apparatus containing the composite specimen in the oven for 4 hr. at 37  $\pm$  2°c. (100  $\pm$  4°F.).
- 5.3 Separate the specimen and the undyed cloth, and dry them apart in air at a temperature not exceeding 60°C. (140°F.).
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloth with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical rating for colour change and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 The Perspirometer developed by the American Association of Textile Chemists and Colorists is intended for carrying out tests of this kind, but any apparatus giving identical results may be used. The "plate method" in the Second Report gives identical results. If it is to be used, §§ 5.1 and 5.2 should be replaced by the following—
  - 5.1 Thoroughly wet the composite specimen in the solution (see § 3.3) at room temperature. Lay it out smooth in a flat-bottomed dish (area of bottom greater than 6.5 cm. × 6.5 cm.) and cover it with the solution. Place a smooth glass plate (5 cm. × 5 cm. or slightly larger weighing approx. 50 g.) on it and press evenly and lightly with the fingers to remove air bubbles. Allow to stand for 15 min. at room temperature.
  - 5.2 Pour off the solution without removing the glass plate. Allow the specimen to remain under the plate for 4 hr. at 37 ± 2°C. (100 ± 4°F.).
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Water Spotting

(PROPOSED ISO RECOMMENDATION)

Note-This test replaces the test for Water Spotting in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to spotting by water.

#### 2. Principle

2.1 A droplet of distilled water is worked into the textile with a glass rod, and the change in colour of the textile is assessed with a standard grey scale after 2 min. and after drying.

#### 3. Apparatus and Reagents

- 3.1 Pipette or dropper to deliver approx. 0.15 ml. of water.
- 3.2 Glass rod with rounded end
- 3.3 Grey Scale for assessing Change in Colour (see §7.1).

#### 4. Specimen

4.1 If the textile to be tested is fabric, a piece at least 10 cm. × 4 cm. is required.

- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece at least 10 cm. × 4 cm., or make a wick of parallel lengths 10 cm. long and about 0.5 cm. in diameter, tied near both ends.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet at least 10 cm. × 4 cm. in size.

#### 5. Procedure

- 5.1 Spot the specimen with approx. 0·15 ml. of distilled water at room temperature. Work the droplet into the specimen with the glass rod.
- 5.2 After 2 min. assess the change in colour in the periphery of the spot with the grey scale.
- 5.3 Dry the specimen in air at room temperature, and assess the change in colour with the grey scale.

#### 6. Report

6.1 Report the numerical ratings for change in colour after 2 min. and after drying.

#### 7. Notes

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing (especially §9.5)
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

#### ISO TENTATIVE TESTS

#### Colour Fastness to Alkaline Milling

(ISO TENTATIVE TEST)

Note— This test replaces the test for Alkaline Milling which was under revision in the Second Edition of the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of wool and of textiles containing wool to alkaline milling.

#### 2. Principle

2.1 A specimen of the textile along with specified undyed cloths is made into a bag containing two stainless-steel balls, and the composite specimen is treated in the Wash Wheel or equivalent apparatus in a solution containing soap and sodium carbonate. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Suitable container with means of agitation (see §7.1)
- 3.2 Stainless-steel balls, 1-9 cm. in diameter
- 3.3 Two undyed cloths each 8 cm. × 8 cm. for testing cloth and yarn or 16 cm. × 8 cm. for testing loose fibre, one piece made of the same kind of fibre as that of the textile to be tested, the second made of a fibre as indicated below or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is—	Second Piece to be-
Cotton	Wool	Acetate rayon	Wool
Wool	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool
Viscose rayon	Wool		

3.4 Solution containing 50 g. soap and 10 g. anhydrous sodium carbonate per litre. The soap must contain not more than 5% moisture and comply with the following specifications based upon dry weight\*—

Free alkali calculated as Na <sub>2</sub> CO <sub>2</sub>	0.3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty saids presented from the soon	30°c maximum

<sup>\*</sup> Soap complying with B.S. 1912: 1953 is suitable for use in this test

- 3.5 Test Control- A dyeing of 3% Disulphine Blue ANS (C.I. 673) on wool cloth
- 3.6 Grey scales for assessing change in colour and staining of undyed cloths (see §7.3).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, a specimen 16 cm. × 8 cm. is required. Sew each undyed cloth (see §3.3) on to the coloured sample along its four edges and in addition in one direction only at intervals of approx. 1 cm. Weigh the composite specimen, fold to form a square with the undyed cloths inside, and sew along two outer edges. Introduce two stainless-steel balls (see §3.2) into the bag so formed, and sew along the remaining outer edge.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress an amount at least equal in weight to the undyed wool. Lay the loose fibre evenly between the undyed cloths each 16 cm. × 8 cm., and sew the layers together by stitching in one direction at intervals of approx. 1 cm. Weigh the specimen, fold to form a square with the undyed wool outside, and treat as in §4.1.

#### 5. Procedure

- 5.1 Carry out the operations described in §§5.2-5.6 inclusive with the composite specimen and the composite test-control specimen in parallel in separate baths.
- 5.2 Put the composite specimen into the metal pot of the Wash Wheel (see §7.1) with three times its own weight of the milling solution (see §3.4). Run the Wash Wheel for 2 hr. at 40°c. (104°F.).
- 5.3 Add sufficient water at 40°C. (104°F.) to give a liquor ratio of 100: 1, and run the Wash Wheel for a further 10 min.
- 5.4 Remove the composite specimens, rinse for 10 min. in cold running tap-water, separate the specimens, and dry them at a temperature not exceeding 60°C. (140°F.).
- 5.5 Assess the effect on the test control with the grey scales. If the change in colour and the staining of undyed materials are not equal to 3 on the appropriate grey scale, the test has not been carried out correctly, and the operations described in §§ 5.1-5.6 must be repeated with fresh composite specimens and a fresh composite test-control specimen.
- 5.6 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.3).

#### 6. Report

6.1 Report the numerical rating for change in colour of the specimen and the numerical rating for staining of each kind of undyed fibre tested.

#### 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists, the Launder-Ometer sponsored by the American Association of Textile Chemists and Colorists, or other mechanical apparatus giving identical results may be used for the test. The Wash Wheel can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 Test Control—A well wetted-out pattern of wool cloth is entered at 40°c. into a dyebath containing 3% Disulphine Blue ANS, 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O), and 3% sulphuric acid (sp. gr. 1·84), all percentages being calculated on the weight of wool. A liquor ratio of 40:1 is used. The dyebath is raised to the boil in 30 min., and boiled for 45 min. The pattern is removed, rinsed, and dried.
- 7.3 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Acid Felting: Mild

(ISO TENTATIVE TEST)

Note- The Second Report did not contain a test for Acid Felting

#### 1. Purpose and Scope

1.1 This is intended as a mild method for assessing the resistance of the colour of textiles to the action of hot very dilute mineral acids, as used in the hat-making and felt industries.

The method applies to all kinds of fibre at all stages of treatment.

#### 2. Principle

2.1 A specimen of the textile in contact with undyed fabrics is milled in acid solution, rinsed, and dried. The change in colour of the specimen and the staining of the undyed fabrics are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 A suitable receptacle and a glass rod, flattened at one end, or appropriate mechanical apparatus
- 3.2 Concentrated sulphuric acid (sp. gr. 1-84)
- 3.3 Two rectangular pieces of cloth (10 cm. × 4 cm.), one of which is of wool and the other of another fibre on which it is wished to assess staining. These should be of plain weave, undyed, unsized, and weighing about 125 g./sq. metre
- 3.4 Standard grey scales (see §7.1).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. in size between the pieces of undyed cloth (see §3.3) and stitch through the three thicknesses along four sides.
- 4.2 If yarn is being tested, knit it into fabric or form a layer of parallel lengths of it, and prepare a composite specimen as under §4.1.
- 4.3 If loose fibre is being tested, this should be combed and compressed so as to obtain a layer 10 cm. × 4 cm., which is dealt with as in §4.1.

#### 5. Procedure

- 5.1 A solution is used containing 1 ml. sulphuric acid (sp. gr. 1.84) per litre, at a temperature of 60 ± 2°c. for a period of 1 hr. The liquor ratio is 40:1.
- 5.2 Milling is carried out by hand or, if possible, mechanically. In the case of hand milling, move the specimen about continuously with the glass rod while it is in the milling bath, and press it with the rod every 2 min. without removal from the bath.
- 5.3 For mechanical milling, different types of apparatus are available. These should be set to give the same milling effect as achieved by hand operation.
- 5.4 After milling is completed, rinse the specimen for 10 min. in cold running tap-water and drain it.
  Open out the specimen by breaking the stitching on three sides, and dry in air at a temperature not exceeding 60°C. (140°F.), without allowing the parts of the composite specimen to come into contact.

#### 6. Report

Give the numerical ratings for the change in colour of the specimen and for the staining of the undyed fabrics.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Acid Felting: Severe

(ISO TENTATIVE TEST)

Note- The Second Report did not contain a test for Acid Felting

1. Purpose and Scope

1.1 This is intended as a severe method for assessing the resistance of dyed wool and fur textiles in the loose state or in the half-planked state to the action of acids used in the acid planking process.

2. Principle

2.1 A specimen of the textile along with specified undyed material where appropriate is treated in solutions of acetic acid and sulphuric acid, rinsed, and dried. The change in colour of the specimen and the staining of the undyed textiles are assessed with standard grey scales.

3. Apparatus and Reagents

- 3.1 Solution containing 1 ml. sulphuric acid (sp. gr. 1.84) per litre
- 3.2 Solution containing 5 ml. acetic acid (30%) per litre
- 3.3 Standard grey scales (see §7.1).

4. Specimen

- 4.1 If the material is half-planked felt, a specimen weighing 2 g. is required.
- 4.2 If the material is loose wool or fur, comb and compress a 2-g. specimen and tie it loosely by string with an equal weight of the appropriate undyed textile.

5. Procedure

- 5.1 If the textile to be tested is fur, it is treated in both sulphuric acid (see §3.1) and acetic acid (see §3.2) as separate tests. If the textile to be tested is wool, it is treated in sulphuric acid only (see §3.1).
- 5.2 Bring the test solution to  $90 \pm 2^{\circ}$ c. (194  $\pm 4^{\circ}$ F.). Immerse the specimen in the solution for 30 min., maintaining this temperature. The liquor ratio is 40:1.
- 5.3 Rinse the material in cold running tap-water and dry at a temperature not exceeding 60°C. (140°F.).
- 5.4 Assess the change in colour of the specimen and the staining of undyed materials used in the test with the grey scales (see §7.1).

6. Report

6.1 Report the numerical ratings for colour change and for staining of the undyed fibre tested,

7. Note

- 7.1 See the following sections—
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### Colour Fastness to Cross-dyeing: Wool

(ISO TENTATIVE TEST)

Note-This test replaces the test for Cross Dyeing (Wool) in the Second Report

1. Purpose and Scope

1.1 This method is intended for assessing the resistance of coloured textiles to processes used for dyeing wool.

2. Principle

2.1 Specimens of the textile to be tested along with the undyed fibres of interest are treated in different types of wool dyebath but without any dye. The change in colour of the specimen and the staining of undyed fibres are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Suitable dye vessels
- 3.2 Acetic acid (30%)
- 3.3 Sulphuric acid (sp. gr. 1-84)

- 3.4 Sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O)
- 3.5 Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>2</sub>)
- 3.6 Two undyed cloths each 10 cm. × 4 cm., one made of the same kind of fibre as that of the textile to be tested, the second made of a fibre indicated below or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is—	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Wool
Silk	Wool	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.7 Grey scales for assessing change in colour and staining of undyed cloths (see §7.1).

#### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. in size between the two pieces of undyed cloth (see §3.6), and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 10 cm. × 4 cm., place the sheet between the two undyed cloths, and treat as in §4.1.
- 4.4 Prepare five composite specimens in the manner described.

#### 5. Procedure

- 5.1 Neutral Cross-dyeing—Enter one composite specimen into a bath containing 20% sodium sulphate crystals. Raise the bath to  $98 \pm 2^{\circ}$ C. ( $208 \pm 4^{\circ}$ F.) in 30 min., and keep at this temperature for 90 min.
- 5.2 Acetic Acid Cross-dyeing— Enter one composite specimen into a bath containing 5% acetic acid (30%) and 20% sodium sulphate crystals. Heat the bath to 98 ± 2°C. (208 ± 4°F.) in 30 min., and keep at this temperature for 90 min.
- 5.3 Sulphuric Acid Cross-dyeing— Enter one composite specimen into a bath containing 20% sodium sulphate crystals and 4% sulphuric acid (sp. gr. 1.84). Heat the bath to 98 ± 2°c. (208 ± 4°F.) in 30 min., and keep at this temperature for 90 min.
- 5.4 Acetic Acid—Chrome Cross-dyeing— Enter one composite specimen into a bath containing 20% sodium sulphate crystals and 5% acetic acid (30%). Heat the bath to 98 ± 2°C. (208 ± 4°F.) in 30 min., and maintain at this temperature for 30 min. Add 2% potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), and maintain the bath at 98 ± 2°C. for a further 60 min.
- 5.5 Sulphuric Acid-Chrome Cross-dyeing Enter one composite specimen into a bath containing 20% sodium sulphate crystals and 3% acetic acid (30%). Heat the bath to 98 ± 2°c. (208 ± 4°F.) in 30 min., and maintain at this temperature for 30 min. Add 2% sulphuric acid (sp. gr. 1·84), and maintain the bath at 98 ± 2°c. for a further 15 min. Add 2% potassium dichromate, and maintain at 98 ± 2°c. for a further 60 min.
- 5.6 Rinse the composite specimens in running tap-water, separate the specimens and the undyed cloths, and dry them apart at a temperature not exceeding 60°c. (140°F.).
- 5.7 Assess the change in colour of the specimen and the staining of undyed cloths with the grey scales (see §7.1).
- 5.8 In carrying out the procedures described in §§5.1-5.5 inclusive a liquor ratio of 50: 1 based on the total weight of the specimen is used. The percentages indicated are calculated on the weight of the wool, silk, polyamide, or similar fibre in the composite specimen.

#### 6. Report

6.1 Report the numerical ratings for change in colour and the numerical ratings for staining of each kind of undyed fibre tested.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.



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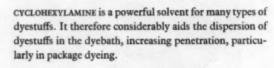
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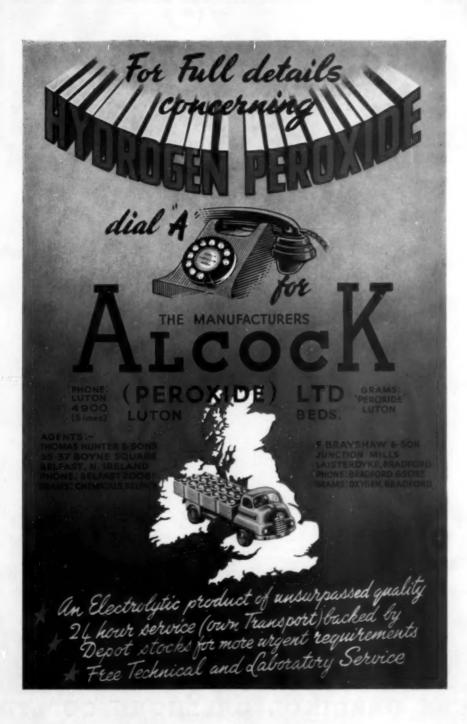
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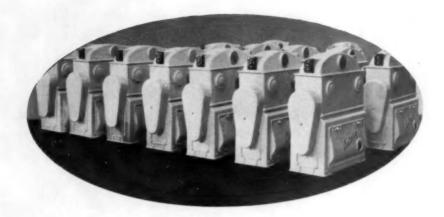
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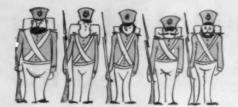
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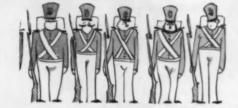


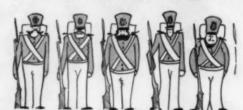




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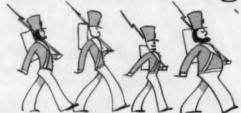
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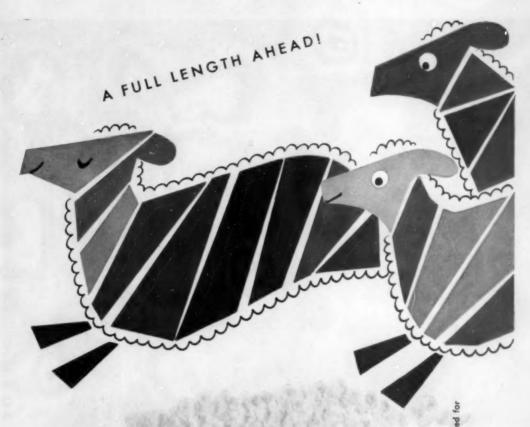


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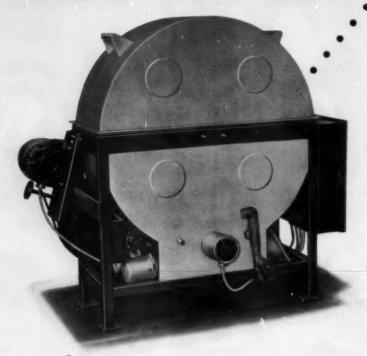
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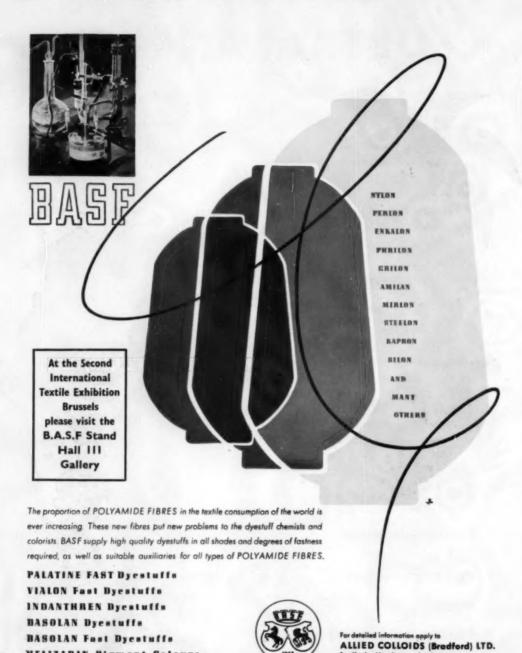
## PLUS

\* Excellent dyeing and levelling properties, covering damage and inequalities in the wool.



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## Colour Fastness to Decatising

(ISO TENTATIVE TEST)

Note- This test replaces the test for Deca.ising in the Second Report

I. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles to the action of steam as employed in the decatising of wool fabrics. Two tests—mild and severe—are given.

2. Principle

2.1 A specimen of the textile is wrapped round a perforated cylinder, and steam is passed through it for 15 min. The specimen is then dried, and the change in colour assessed with a standard grey scale. A test-control fabric is used.

## 3. Apparatus and Reagents

- 3.1 Suitable decatising apparatus (see §7.1)
- 3.2 Test Control—A dyeing of Cloth Fast Blue R (C.I. 288) (see §7.2)
- 3.3 Grey Scale for assessing Change in Colour (see §7.3).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, a specimen of it 10 cm. × 4 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it, place between two pieces of undyed cotton cloth, and sew around the four sides to hold the yarn in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm.
  × 4 cm., place it between two pieces of undyed cotton cloth, and sew around the four sides to hold the fibre in place.
- 4.4 Test Control—Prepare a specimen of the test-control dyeing (§7.2) in the way outlined for cloth in §4.1.

#### 5. Procedure

- 5.1 Carry out the operations described in §§5.2-5.4 inclusive with the specimens and the testcontrol specimen together.
- 5.2 A length of cotton blanket material weighing about 400 g./sq. metre (12 oz./sq.yd.) and napped on both sides is wrapped three times round the perforated cylinder of the decatising apparatus (see §7.1). The specimen is then placed round the wrapped cylinder and covered with three further wrappings of the blanket cloth.
- 5.3 Water-free steam is then passed through the specimen for 15 min. at the following pressures— Mild Decatising— 1.5 kg./sq.cm. (21.3 lb./sq.in.) or 0.5 kg./sq.cm. (7.1 lb./sq.in.) above atmospheric pressure.

Severe Decatising—2.5 kg./sq.cm. (35.5 lb./sq.in.) or 1.5 kg./sq.cm. (21.3 lb./sq.in.) above atmospheric pressure.

- 5.4 Dry the specimen in air at a temperature not exceeding 60°C. (140°F.).
- 5.5 Assess the effect on the test control with the grey scale. If the change is not equal to the following—

Mild Decatising Severe Decatising 4 2 YD

the test has not been carried out correctly, and the operations described in §§5.1-5.6 inclusive must be repeated with fresh specimens and a fresh test-control specimen.

5.6 Assess the change in colour with the grey scale (see §7.3).

#### 6. Report

6.1 Report the numerical rating for change in colour of the specimen, specifying the severity of the test used, viz. mild or severe.

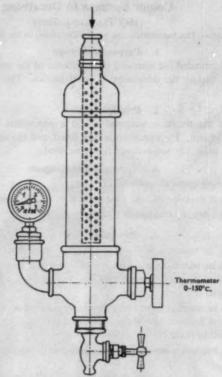


Fig. 3 — Decatising Apparatus

#### 7. Notes

- 7.1 Decatising Apparatus—The apparatus shown in Fig. 3 or similar apparatus shall be used.
- 7.2 Test Control— A well wetted-out pattern of wool cloth is treated at 40-50°C. (104-122°F.) in a bath containing 10% sodium sulphate crystals (Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O) and 4% ammonium acetate (CH<sub>3</sub>·COONH<sub>4</sub>) for 10-15 min. at a liquor ratio of 40:1. 2% Cloth Fast Blue R is then added, the dyebath is brought to a gentle boil in 30 min., and gentle boiling is continued for 45-60 min. The pattern is then removed, rinsed, and dried.
- 7.3 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

## Colour Fastness to Chlorinated Water

(ISO TENTATIVE TEST)
NOTE— This is a new test

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to chlorinated water as used in the modern treatment of water for swimming baths (break-point chlorination).

#### 2. Principle

- 2.1 Specimens of the textile under test are agitated in chlorinated water at different pH values.
- 2.2 An assessment is made on the pattern showing the largest change in colour with the standard grey scale.

## 3. Apparatus and Reagents

- 3.1 Glass beakers or suitable containers with means of agitation
- 3.2 Solution set at pH 6.0 and containing 25 p.p.m. free chlorine (see §§7.1 and 7.4)
- 3.3 Solution set at pH 7.0 and containing 25 p.p.m. free chlorine (see §§7.2 and 7.4)
- 3.4 Solution set at pH 8.5 and containing 25 p.p.m. free chlorine (see §§7.3 and 7.4)
- 3.5 Boric acid (HaBOa)
- 3.6 Sodium hydroxide (NaOH)
- 3.7 Potassium chloride (KCl)
- 3.8 Potassium dihydrogen phosphate (KH<sub>2</sub>PO<sub>4</sub>)
- 3.9 Disodium hydrogen phosphate (Na<sub>2</sub>HPO<sub>4</sub>)
- 3.10 Sodium hypochlorite of the following composition shall be used-

Sodium hypochlorite	NaOCI	14-16%
Sodium chloride	NaCl	12-17%
Sodium hydroxide	NaOH	2-0% maximum
Sodium carbonate	Na,COa	2-0% maximum
Iron	Fe	0-001% maximum

3.11 Grey Scale for assessing Change in Colour (see §7.5).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, three pieces each 10 cm. × 10 cm. in size are required.
- 4.2 If the textile to be tested is yarn or thread, knit it into fabric in accordance with §4.1.

## 5. Procedure

- 5.1 A specimen of the textile is immersed in each of the three preset solutions at room temperature for 1 hr. at a liquor ratio of 100:1.
- 5.2 The specimens are drained, and dried in air at 25  $\pm$  2°C. (77  $\pm$  4°F.) without rinsing.
- 5.3 The operations in §§5.1 and 5.2 are repeated to give a total of five treatments.
- 5.4 Assess with the grey scale the change in colour of the specimen showing the greatest change in colour.

## 6. Report

6.1 Report the numerical rating of the specimen showing the greatest change in colour.

#### 7. Notes

- 7.1 Solution at pH 6·0 containing 100 ml. of m./15 disodium hydrogen phosphate and 900 ml. of m./15 potassium dihydrogen phosphate per litre. Solution to contain 25 p.p.m. free chlorine (see §7.4).
- 7.2 Solution at pH 7·0 containing 600 ml. of M./15 disodium hydrogen phosphate and 400 ml. of M./15 potassium dihydrogen phosphate per litre. Solution to contain 25 p.p.m. free chlorine (see §7.4).
- 7.3 Solution at pH 8.5 containing 5.1 ml. of M./10 sodium hydroxide, 0.0144 g. of potassium chloride, and 0.1123 g. of boric acid per litre. Solution to contain 25 p.p.m. free chlorine (see §7.4).
- 7.4 Sodium hypochlorite should be analysed for chlorine content by a standard technique (with iodine and thiosulphate) and diluted to 25 p.p.m., preferably in two stages, e.g.—
  - (a) 2 g./litre = 13.3 ml. of sodium hypochlorite containing 150 g. chlorine per litre, diluted to a litre
  - (b) 25 p.p.m. = 12.5 ml. of solution (a) per litre.
- 7.5 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour.

## Colour Fastness to Degumming

(ISO TENTATIVE TEST)

NOTE—This test replaces the test for Degumming in the Second Report

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds to the action of soap liquors used in removing natural gum from silk.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is boiled in a solution of soap, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Conical flask with a water-cooled finger condenser fitting loosely in the neck
- 3.2 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na <sub>2</sub> CO <sub>3</sub>	0-3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty acids prepared from the soap	30°C. maximum

\* Soap complying with B.S. 1912: 1953 is suitable for use in this test

- 3.3 Solution containing 15 g. of soap (see §3.2) per litre
- 3.4 An undyed cloth 10 cm. × 4 cm. made of undegummed cultivated silk fabric
- 3.5 A second cloth, 10 cm. × 4 cm., made of another fibre may also be used if the staining on such other fibre is required
- 3.6 Grey scales for assessing change in colour and staining of undyed cloths (see §7.1).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, a specimen 10 cm. × 4 cm. is required. Place the undyed silk cloth (see §3.4) over the specimen, and sew along all four sides to form a composite specimen. If another fibre is involved (see §3.5), place the second undyed cloth on the opposite side of the specimen to that occupied by the undyed silk cloth, and sew along all four sides to make a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between two undyed cloths and sew around the four sides to hold the yarn in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between two undyed cloths, sew around the four sides to hold the fibres in place, and treat as in §4.1.

#### 5. Procedure

- 5.1 Treat the composite specimen in the boiling solution of soap (see §3.3) at a liquor ratio of 100: 1 for 2 hr. under reflux.
- 5.2 Rinse the composite specimen thoroughly in cold distilled water, separate the components, and dry.
- 5.3 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.1).

## 6. Report

6.1 Report the numerical ratings for colour change and for staining of the undyed silk and of the second undyed cloth if used.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Steaming

(ISO TENTATIVE TEST)

NOTE- This test replaces the test for Steaming in the Second Report

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of acetate rayon in all forms to steaming in processing.

2. Principle

2.1 A specimen of the acetate rayon and undyed acetate rayon and cotton cloths are rolled into a cylinder, and placed in the neck of a flask containing boiling water. The colour which is transferred to the acetate rayon is assessed with a standard grey scale.

## 3. Apparatus and Reagents

- 3.1 Flask with long neck
- 3.2 Undyed scoured acetate rayon cloth
- 3.3 Undyed bleached cotton cloth
- 3.4 Standard Grey Scale for assessing Staining (see §7.1).

## 4. Specimen

- 4.1 A piece of the undyed scoured acetate rayon cloth 10 cm. × 4 cm. is placed between two pieces of undyed bleached cotton cloth of the same size. If the specimen is fabric, place it on one of the pieces of cotton to form a composite sample.
- 4.2 If the textile to be tested is yarn, knit it into fabric, and treat a piece at least 10 cm. × 4 cm. as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between two undyed scoured cotton cloths, and treat as in §4.1.

#### 5. Procedure

- 5.1 Bring water in the flask to the boil. Roll the composite sample into the form of a cylinder with the specimen inside the roll, wrap the cylinder in felt, so that the whole is an easy fit in the neck of the flask, and boil for 30 min.
- 5.2 Assess the staining of the undyed acetate rayon cloth with the Grey Scale for assessing Staining (see §7.1).

## 6. Report

6.1 Report the numerical rating for staining of the undyed acetate rayon cloth.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Staining.

## **Colour Fastness to Sublimation**

(ISO TENTATIVE TEST)

Note- This replaces the test for Storage in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles to sublimation during storage. It is mainly applicable to acetate rayon.

## 2. Principle

2.1 A specimen of the acetate rayon fabric in contact with undyed scoured acetate rayon cloth is rolled into a cylinder, and subjected to dry heat. The colour which sublimes on to the undyed acetate rayon cloth is assessed with a standard grey scale.

## 3. Apparatus and Reagents

- 3.1 A glass tube about 15 mm. in diameter
- 3.2 Oven for treating composite specimens at  $120 \pm 2^{\circ}$ C.  $(248 \pm 4^{\circ}$ F.)
- 3.3 Undyed scoured acetate rayon cloth -
- 3.4 Grey Scale for assessing Staining (see §7.1).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. on undyed scoured acetate rayon of the same size, to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric, and treat a piece 10 cm. × 4 cm. as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between two undyed scoured acetate rayon cloths, and treat as in 84.1.

#### 5. Procedure

- 5.1 Roll the composite specimen into a cylinder, and place in a glass tube 15 mm. in diameter.
- 5.2 Heat the tube and the specimen in an oven at 120°c. (248°F.) for 5 hr.
- 5.3 Remove the specimen from the tube and unroll.
- 5.4 Assess the staining of the undyed acetate rayon with the Grey Scale for Staining (see §7.1).

#### 6. Report

6.1 Report the numerical rating for staining of the undyed acetate rayon cloth.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Staining

## Colour Fastness to Bleaching with Sodium Chlorite

(ISO TENTATIVE TEST)

Note- The Second Report did not contain a test for bleaching with sodium chlorite

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds except wool and silk to the action of bleaching baths containing sodium chlorite in concentrations commonly employed in textile processing. It is mainly applicable to natural and regenerated cellulose, polyamides, and other synthetic fibres.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is treated with sodium chlorite solution, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloth are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Two concentrations of sodium chlorite solution are required (see §7.2)—
- 3.1.1 For regenerated cellulose, polyamide, and other synthetic fibres—
  Sodium chlorite solution containing 1 g. sodium chlorite (80%) per litre, activated with acetic acid: pH 3.5.
- 3.1.2 For natural cellulose-
  - Sodium chlorite solution containing 2.5 g. sodium chlorite (80%) and 0.1 g. sodium dihydrogen pyrophosphate per litre activated with formic acid: pH 3.5.
- 3.2 Pieces of undyed cloth, each 10 cm. × 4 cm.
- 3.3 Test control (see §7.3).
- 3.4 Grey scales for assessing change in colour and staining of undyed cloth (see §7.1).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. on a piece of the undyed cloth (see §3.2), and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat as in §4.1, or form a layer of parallel lengths of it between two pieces of the undyed cloth and sew around the four sides to hold the yarn in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 10 cm. × 4 cm. in size. Place the sheet between two pieces of the undyed cloth, and sew along the four sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Wet out the specimen in the test solution and immerse in the bleaching bath (see §3.1.1 or 3.1.2) for 1 hr. at  $80 \pm 2^{\circ}$ c. (176  $\pm 4^{\circ}$ F.) at a liquor ratio of 50:1, without agitation.
- 5.2 Remove the specimen and rinse it for 10 min. in cold running tap-water. Remove the stitching from two long sides and one short side of the composite specimen, open it out, and dry at a temperature not exceeding 60°C. (140°F.) with the three parts in contact only at the remaining line of stitching.
- 5.3 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.1).

## 6. Report

6.1 Report the bleach solution used, the numerical rating for colour change, and the numerical rating for staining of the undyed cloth.

## 7. Notes

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness of Textiles: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness of Textiles: Grey Scale for assessing Staining.
- 7.2 The concentration of sodium chlorite employed is determined by titration with sodium thiosulphate—

1 ml. of 0·1 m.thiosulphate solution = 0·00226 g. NaClO<sub>2</sub>

7.3 Unbleached cleaned cotton cloths may be used as test-control specimens. The degree of whiteness shown after the bleaching process indicates the effect of the test.

## Colour Fastness to Washing: Mechanical Washing A

(ISO TENTATIVE TEST)

Note— This test replaces Washing Test No. 2 for Natural and Regenerated Cellulose and Washing Test No. 3 for Wool in the Second Report

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the action of soap and soda solutions as used in mechanical washing processes. It is mainly intended to be used as a severe test for wool textiles and as an intermediate test for cellulosic textiles.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a solution containing soap and sodium carbonate in the Society's Wash Wheel, the AATCC Launder-Ometer, or alternative apparatus giving the same result; the specimen is then rinsed and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Suitable mechanical washing device (see §7.1)
- 3.2 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na<sub>2</sub>CO<sub>2</sub>

Free alkali calculated as NaOH

Combined fatty acids calculated as Na salts

Titre of mixed fatty acids prepared from the soap

30°C. maximum

30°C. maximum

\* Soap complying with B.S. 1912: 1953 is suitable for use in this test

- 3.3 Soap solution, containing 5 g. of soap (see §3.2) and 2 g. of anhydrous sodium carbonate per litre of distilled water
- 3.4 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below or as otherwise specified—

If First Piece is— Cotton	Second Piece to be— Wool	If First Piece is— Viscose rayon	Second Piece to be
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.5 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of undyed cloth required in §3.4, and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm.
  × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

#### 5. Procedure

- 5.1 Place the composite specimen in the container and add the necessary amount of soap and soda solution, previously heated to  $60 \pm 2^{\circ}$ c. (140  $\pm 4^{\circ}$ F.), to give a liquor ratio of 50:1.
- 5.2 Treat the composite specimen for 30 min. at  $60 \pm 2^{\circ}$ c.  $(140 \pm 4^{\circ}$ F.).
- 5.3 Rinse the composite specimen twice in cold distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not above 60°C. (140°F.) with the three parts in contact only along the one row of stitching.
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

## 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists, the Launder-Ometer sponsored by the American Association of Textile Chemists and Colorists, or other mechanical apparatus giving identical results may be used for the test. The Wash Wheel can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Washing: Mechanical Washing B

(ISO TENTATIVE TEST)

Note- This test replaces Washing Test No. 3 for Natural and Regenerated Cellulose in the Second Report

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles which are physically capable of withstanding the action of soap solutions near the boiling point. It is mainly intended to be used as a severe test for cellulosic fibres.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a solution containing soap and sodium carbonate in the Society's Wash Wheel, the AATCC LaunderOmeter, or alternative apparatus giving the same result; the specimen is then rinsed and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Suitable mechanical washing device (see §7.1)
- 3.2 Monel-metal or stainless-steel balls approx. 0.6 cm. (1 in.) in diameter
- 3.3 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na <sub>2</sub> CO <sub>3</sub>	0.3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85 % minimum
Titre of mixed fatty acids prepared from the soap	30°c, maximum

\* Soap complying with B.S. 1912: 1953 is suitable for use in this test

- 3.4 Soap solution, containing 5 g. of soap (see §3.3) and 2 g. anhydrous sodium carbonate per litre of distilled water
- 3.5 Undyed cloths of the type under test (or, if fibre or yarn is being tested, undyed cloth made from the same kind of fibre)
- 3.6 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, sew a specimen of it 10 cm. × 4 cm. to an equal-sized piece of the undyed cloth (see §3.5) along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between two pieces of undyed cloth and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm.
  × 4 cm., place it between two pieces of undyed cloth, and sew around the four sides to hold the fibre in place.

## 5. Procedure

- 5.1 Place the composite specimen in the container together with 10 balls as specified in §3.2, and add the necessary amount of soap and soda solution, previously heated to 95 ± 2°c. (203 ± 4°r.), to give a liquor ratio of 50: 1.
- 5.2 Treat the composite specimen for 30 min. at 95  $\pm$  2°c. (203  $\pm$  4°F.).
- 5.3 Rinse the composite specimen twice in cold distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not exceeding 60°c. (140°F.) with the three parts in contact only along the one row of stitching.
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.2).

## 6. Report

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

## 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists, the Launder-Ometer sponsored by the American Association of Textile Chemists and Colorists, or other mechanical apparatus giving identical results may be used for the test. The Wash Wheel can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Washing at the Boil

(ISO TENTATIVE TEST)

Note-The Second Report did not contain a test for Washing at the Boil

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds which are physically capable of withstanding the action of boiling soap solutions, especially those coloured with vat or similar dyes of the best possible fastness, e.g. linen borders, trimmings, etc.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is boiled in a solution containing soap and sodium carbonate; the specimen is then squeezed, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Water bath containing sodium nitrate to ensure a temperature of 110°c. (230°F.)
- 3.2 300-ml. beaker of stainless steel, of diameter at bottom 65 mm., covered with a water-cooled overlapping watch-glass
- 3.3 Plate of stainless steel, of diameter 60 mm. and thickness 2 mm., containing 15 holes of diameter 5 mm. equally spaced over the plate, to weight the specimen during boiling
- 3.4 Glass rod flattened at one end
- 3.5 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Pour and monday	
Free alkali calculated as Na <sub>2</sub> CO <sub>3</sub>	0-3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty acids prepared from the soap	30°C. maximum

- \* Soap complying with B.S. 1912: 1953 is suitable for use in this test
- 3.6 Solution of distilled water containing 0.5% soap (see §3.5) and 0.2% anhydrous sodium carbonate
- 3.7 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of another suitable fibre
- 3.8 Grey scales for assessing change in colour and staining of undyed cloths (see §7.1).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between two pieces of undyed cloth, and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarn in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

## 5. Procedure

- 5.1 Treat the specimen for 30 min, in the boiling soap solution at a liquor ratio of 50:1. To prevent the specimen from floating in the soap froth during boiling, place the perforated stainless-steel plate (see §3.3) on the specimen in the beaker, and cover with the watch-glass.
- 5.2 After boiling for 30 min. press the composite specimen 15 times with a glass rod (see §3.4) without removing it from the solution.

- 5.3 Rinse the composite specimen twice in distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not exceeding 60°C. (140°F.), with the three parts in contact only along the one row of stitching.
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloth with the grey scales.

## 6. Report

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

#### 7. Note

- 7.1 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness of Textiles: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness of Textiles: Grey Scale for assessing Staining.

## Colour Fastness to Washing in the Presence of Sodium Hypochlorite

(ISO TENTATIVE TEST)

Note- The Second Report did not contain a test for Washing in the presence of Sodium Hypochlorite

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds and in all forms to washing at high temperature in the presence of a bleaching agent.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a soap solution containing sodium hypochlorite at 83°C. (181°F.) in the Society's Wash Wheel, the AATCC Launder-Ometer, or alternative apparatus giving the same result; the specimen is then rinsed and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Suitable mechanical washing device (see §7.1)
- 3.2 Ten Monel-metal or stainless-steel balls approx. 0.6 cm. in diameter
- 3.3 Hand iron (see §7.2)
- 3.4 Undyed, bleached, unsized cotton cloth weighing approx. 125 g./sq.metre (3.7 oz/sq.yd.) and having approx. 32 threads per centimetre (80 threads per inch) in warp and west
- 3.5 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

on dry weight —	
Free alkali calculated as Na <sub>2</sub> CO <sub>3</sub>	0-3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty saids appared from the sone	30°c movimum

- \* Soap complying with B.S. 1912: 1953 is suitable for use in this test
- 3.6 Solution of distilled water containing 0.5% soap (see §3.5) and 0.2% anhydrous sodium carbonate
- 3.7 Sodium hypochlorite, available chlorine content to be checked at frequent intervals (see §7.3). A small amount is diluted to contain 1% of available chlorine just before use
- 3.8 Acetic acid (28%). A portion is diluted to a concentration of 0.014% when used
- 3.9 Standard grey scales (see §7.4).

## 4. Specimens

4.1 If the textile to be tested is fabric, sew a piece of it 10 cm. × 4 cm. to a 4-cm. square of the undyed cloth (see §3.4) with a single line of stitching near the end of the specimen to make a composite specimen. The undyed cloth is to be in contact with the face of printed material.

- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

## 5. Procedure

- 5.1 Place the composite specimen and ten 0.6-cm. steel balls (see §3.2) in a glass jar (see §7.1) with 100 ml. of the soap solution (see §3.6) at 83°C. (182°F.), to which 1 ml. of sodium hypochlorite solution containing 1% of available chlorine is added immediately before the specimen is placed in it. To avoid breakage of the jar and to attain working temperature more easily it is advisable to preheat the jar and contents in a water bath. Close the jar and place it in the machine (see §7.1), which is at the required temperature, and run the machine for 45 min.
- 5.2 Empty the jar and rinse the specimen twice with 100 ml. of water at 40°C. (104°F.) by shaking it vigorously with the water in the jar for 1 min. each time.
- 5.3 Pour the specimen in 100 ml. of a 0·014% solution of acetic acid (0·05 ml. of 28% acetic acid per 100 ml. of water) by shaking it vigorously in the jar with the acid solution at 27°C. (80°F.) for 1 min.
- 5.4 Rinse the specimen once more in 100 ml. of water at 27°C, (80°F.) for 1 min.
- 5.5 Hydroextract or wring the specimen dry with the undyed cloth on top by pressing with an iron (see §7.2) having a temperature of 135–150°C. (275–302°F.) at the point, except in the case of fabrics known to have a low fusion temperature, in which case the temperature of the iron should be reduced accordingly.
- 5.6 Assess the change in colour of the specimen and the staining of the undyed cloths with standard grey scales (see §7.4).

#### 6. Report

6.1 Report the numerical rating for colour change of the specimen and the numerical rating for staining of the undyed cloth.

## 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists, the Launder-Ometer sponsored by the American Association of Textile Chemists and Colorists, or other mechanical apparatus giving identical results may be used for the test. The Wash Wheel can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 A 5-6 lb. iron with 1000-watt heating unit is recommended in order to avoid large fluctuations in temperature. The temperature of the iron can be determined conveniently with a calibrated thermocouple, a thermometer inserted in a well in the iron, or alloys melting at appropriate temperatures. It must be remembered in using the second method that there is considerable lag between the temperature indicated by the thermometer and the actual surface temperature of the iron; and in using the second and third methods that the iron may be cooled by contact with the cloth. Alloys of tin, lead, and bismuth in the proportions 16:25:16 and 9:8:4 melt at approx. 136°C. (277°F.) and 148°C. (298°F.) respectively. If small particles of these alloys are placed on the iron, one alloy will melt and the other will not if the temperature of the iron is within the indicated range of temperature. Temperature indicators furnished as an integral part of some irons should be calibrated for accuracy.
- 7.3 The pH value and the available chlorine content of commercial brands of sodium hypochlorite (NaOCl) vary from 9.8 to 12.8 and from 4% to 6% respectively. The actual available chlorine should be determined before use. The following method is suggested—

Pipette a 25-ml. sample into a 250-ml. Erlenmeyer flask. Add about 150 ml. of water and 50 ml. of 0·1 N. sodium arsenite (Na<sub>2</sub>HAsO<sub>3</sub>) solution. Titrate the excess of sodium arsenite with 0·1 N. iodine solution. Calculate as Cl.

1 ml. of 0-1 N. sodium arsenite = 0-003546 g. of Cl

Any hypochlorite solution with an initial pH between 11·2 and 12·8 will be buffered to within the desired 10·8–11·2 if diluted to the available chlorine content required for test use with a mixture composed of 5 parts of a 1% solution of sodium bicarbonate (NaHCO<sub>3</sub>) and 95 parts of a 5% solution of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>). However, if the initial pH is between 9·6 and 11·2, 0·5 g. of sodium hydroxide (NaOH) should first be added to each 500 ml., preferably in the form of a 10% solution. This will raise the pH to within the range which the buffer solution will satisfactorily control. This addition of sodium hydroxide will not impair the stability of the hypochlorite solution. Storage in coloured glass bottles, tightly stoppered, in a cool place, protected from strong light, aids stability.

- 7.4 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grev Scale for assessing Staining.

## TESTS UNDER CONSIDERATION BY ISO

## Colour Fastness to Perspiration

(UNDER CONSIDERATION BY ISO)

Note— This test replaces the tests for Perspiration in the Second Report. An account of the investigations into Fastness to Perspiration is given in J.S.D.C., 68, 392 (1952) and 70, 63 (1954)

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to perspiration.

## 2. Principle

2.1 Specimens of the textile in contact with specified undyed cloths are treated in solutions containing histidine and sodium chloride, drained, and placed in a Perspirometer or equivalent apparatus (see §7.1). The specimens and undyed cloths are dried separately. The change in colour of each specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Perspirometer or equivalent apparatus (see §7.1)
- 3.2 Solution containing 5 g. histidine hydrochloride and 5 g. sodium acetate (CH<sub>3</sub>·COONa, 3H<sub>2</sub>O) per litre of distilled water (pH 5-5)
- 3.3 Solution containing 5 g. histidine hydrochloride per litre of distilled water brought to pH 8 by addition of 1-0 N. sodium carbonate
- 3.4 Two undyed cloths, each 5 cm. × 5 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified (see §7.2)—

If First Piece is-	Second Piece to be-	If First Piece is-	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

- 3.5 Standard grey scales (see §7.6)
- 3.6 Oven maintained at  $37 + 2^{\circ}$ C. (100  $\pm 4^{\circ}$ F.).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 5 cm. × 4 cm. (see §7.3) between the two pieces of undyed cloths (see §3.4) so that an area of 5 cm. × 1 cm. of the undyed cloths is not in contact with the specimen, and sew along the common 5-cm. side to form a composite specimen. Two such composite specimens are required for the test. (See §7.3.)
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew along two opposite sides near the ends of the yarns to form a composite specimen. Two such composite specimens are required.
- 4.3 If the textile to be tested is loose fibre, comb and compress some of it into a sheet 5 cm. × 4 cm. in size, place the sheet between the two undyed cloths as in §4.1, and sew along two opposite sides to form a composite specimen. Two such composite specimens are required.

## 5. Procedure

- 5.1 Thoroughly wet one composite specimen in each of the solutions in §§3.2 and 3.3, at a liquor ratio of 50: 1, and allow them to remain in the solutions for 30 min. at room temperature. Pour off the solution and place each composite specimen between two glass plates measuring about 7.5 cm × 6.5 cm. under a force of about 4.5 kg. (10 lb.).
- 5.2 Place the apparatus containing the composite specimens in the oven for 4 hr. at 37  $\pm$  2°c. (100  $\pm$  4°F.).
- 5.3 Separate the specimens and undyed cloths, and dry them apart in air at a temperature not exceeding 60°C. (140°F.).
- 5.4 Assess the change in colour of each of the specimens and the staining of the undyed cloths (see §3.4) with standard grey scales (see §7.6).

## 6. Report

6.1 For each of the solutions specified in §§3.2 and 3.3 report the numerical rating for colour change of the specimen and the numerical rating for staining of each kind of undyed fibre used in the test.

#### 7. Notes

- 7.1 The Perspirometer developed by the American Association of Textile Chemists and Colorists is intended for carrying out tests of this kind, but any apparatus giving identical results may be used. The "plate method" in the Second Report gives identical results. If it is to be used, §§5.1 and 5.2 should be replaced by the following—
  - 5.1 Thoroughly wet one composite specimen in each of the solutions in §§3.2 and 3.3 at a liquor ratio of 50: 1, and allow them to remain in the solution for 30 min. at room temperature. Lay out each composite specimen smooth in a flat-bottomed dish (area of bottom greater than 5 cm. × 5 cm.), and cover each with the corresponding solution. Place a smooth glass plate (5 cm. × 5 cm. or slightly larger weighing 50 g.) on each, and press evenly and lightly with the fingers to remove air bubbles. Allow to stand for 15 min. at room temperature.
  - 5.2 Pour off the solution without removing the glass plates. Allow the composite specimens to remain under the plates for 4 hr. at 37 ± 2°C. (100 ± 4°F.).
- 7.2 See reference (a) in §7.6 below, especially §7.8 in it.
- 7.3 Experience has shown that this method of preparing the composite specimen, which ensures that an area of undyed cloths cannot be stained, considerably facilitates the assessment of staining where this occurs.
- 7.4 It is known that the prescribed solutions will not reproduce the effect of natural perspiration on wool materials dyed with Alizarine Saphirole B (C.I. 1054). As this is due to the action of salt, it is recommended that in appropriate cases a further test should be made for Colour Fastness to Sea Water.

- 7.5 In many cases of cellulosic fibres dyed with direct dyes containing copper or aftertreated with copper salts, the prescribed tests and natural perspiration bring about a removal of copper from the dyeings. This may cause a material alteration in fastness to light and washing, and it is therefore recommended that this possibility should be taken into consideration.
- 7.6 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Burnt Gas Fumes

(UNDER CONSIDERATION BY ISO)

Note— This test replaces the test for Burnt Gas Fumes which was under revision in the Second Edition of the Second Report

## 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the products of combustion of coal-gas.

## 2. Principle

2.1 Specimens of the textile under test are agitated in a current of burnt coal-gas and air. The change in colour of the specimen is assessed with the standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Burnt gas fume apparatus (see §7.3)
- 3.2 Test control (see §7.1)
- 3.3 Grey Scale for assessing Change in Colour (see §7.2).

#### 4. Specimens

- 4.1 If the textile to be tested is fabric, a piece 4 cm. × 5 cm. is required.
- 4.2 If the textile to be tested is yarn, knit it into fabric and use a piece 4 cm. × 5 cm.

## 5. Procedure

- 5.1 Condition the specimen to be tested at a relative humidity of 65  $\pm$  2% and at a temperature of 20  $\pm$  2°c. (68  $\pm$  4°F.).
- 5.2 Mount specimens on the frame of the burnt gas fume apparatus (see §7.3) together with the test-control specimens (see §7.1).
- 5.3 Fill blank spaces on the frame of the apparatus with undyed acetate rayon fabric.
- 5.4 Position the bell-jar and connect to a source of burnt gas fumes.
- 5.5 Warm the bell-iar to  $25 + 2^{\circ}$ C.  $(77 + 4^{\circ}$ F.).
- 5.6 Ignite the micro-burner and adjust so that the gas-air mixture contains 5.0% of gas. Suitable amounts are 6 litres of total gaseous product and 300 c.c. of gas per minute.
- 5.7 Adjust the speed of rotation of the frame to 70-80 r.p.m.
- 5.8 Expose the specimens until the principal test control shows a contrast similar to Grade 2 on the grey scale.
- 5.9 Treat exposed specimens for 5 min. at 25°C. (77°F.) in 1 % triethanolamine solution, squeeze, and dry.
- 5.10 Assess the change in colour with the grey scale (see §7.2).

#### 6. Report

6.1 Report the numerical rating for change in colour.

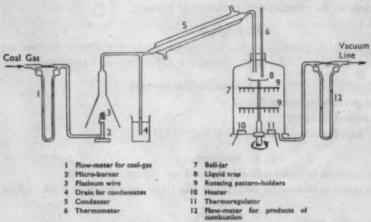
## 7. Notes

## 7.1 Test Control Specimens-

- 7.1.1 Immerse the acetate rayon in 2.0% Duranol Blue 2G 300 (ICI) dye set with 1 part of soap per 1000 parts of water, at a liquor ratio of 50: 1. Commence dyeing at 25 ± 2°C. (77 ± 4°F.) and slowly raise to 85°C. (185°F.). Continue dyeing at 85°C. (185°F.) for 45 min. Hydroextract and dry at a temperature not exceeding 100°C. (212°F.).
- 7.1.2 The auxiliary test control is dyed similarly with 0.8% Artisil Direct Blue GFL (S).

## 7.2 See the following sections—

- (a) Colour Fastness of Textiles: General Principles of Testing
- (b) Colour Fastness: Grey Scale for assessing Change in Colour.
- 7.3 Burnt Gas Fume Apparatus— The apparatus (Fig. 4) consists essentially of rotating arms on which the patterns are hung, the whole being enclosed in a bell-jar of 8-12 litres capacity. A mixture of burnt coal-gas (from a micro-burner) and air is drawn through the bell-jar at a metered rate by means of a vacuum line or water pump. This part of the apparatus consists



Fro. 4 - Burnt Gas Fume Apparatus

of two flow-meters (the first for coal-gas, the second for total gaseous products), a coal-gas micro-burner, a funnel to collect all the products of combustion, and a lead to the top of the bell-jar. In this lead is incorporated a device for dissipating some of the heat of the combustion of the coal-gas and for removing the greater portion of the moisture present in the burnt gas fumes. This can take the form of a water-cooled Liebig condenser or similar apparatus. An exit tube through the plate glass base of the bell-jar is connected to the vacuum line through the second flow-meter. A 25-watt electric bulb (or similar source of heat) may be used to warm the bell-jar and its contents to the defined testing temperature of  $25 \pm 2^{\circ}$ C. (77  $\pm 4^{\circ}$ F.). A length of platinum wire is attached to the micro-burner and adjusted in spiral form to be in the Bunsen flame. It has been found that fading is accelerated by the presence of the platinum wire in the flame.

The flow-meters and the micro-burner have the following specifications-

## (a) FLOW-METERS-

- (1) Coal-gas—to read 0-0.5 litre/min.
- (2) Air—to read 0-10 litres/min.
- (b) BUNSEN MICRO-BURNER— Brass tube of diameter 0.5 cm. (3 in.) with regulator.

It has been found that an exposure of 8 hr. is usually adequate for producing the change of colour of the test control similar to Grade 2 on the grey scale. For convenience an auxiliary control may also be used as Grade 4 test control, and this has been found to give a change of colour under the same exposure equal to that illustrated by Grade 4 on the grey scale.

During preferred conditions of working there is a small condensation of moisture on the upper surfaces of the bell-jar, but no appreciable condensation on the patterns or on the lower surfaces of the bell-jar.

## RESIDUAL SECOND-REPORT TESTS

## Colour Fastness to Washing: S.D.C. No. 2

Note— This test is a modification of the Washing Test No. 2 for Wool in the Second Report. It differs from it in the following respects—

- (a) Preneutralisation is omitted.
- (b) The soap concentration is increased from 2 to 5 g./litre.
- (c) Assessments are now made with grey scales and not on a pass-fail basis.

## 1. Purpose and Scope

1.1 This test is intended for assessing the resistance of the colour of textiles of all kinds in all forms to the repeated action of soap solution as used in washing them by hand.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a soap solution, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

- 3.1 Wash Wheel (see §7.1)
- 3.2 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na.CO.	0-3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty acids prepared from the soap	30°C. maximum

- \* Soap complying with B.S. 1912: 1953 is suitable for use in this test
- 3.3 Soap solution, containing 5 g. of soap (see §3.2) per litre of distilled water
- 3.4 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is-	Second Piece to be-
Cotton	Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Viscose rayon
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.5 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

### 4. Specimen

- 4.1 If the textile to be tested is fabric, place a specimen of it 10 cm. × 4 cm. between the two pieces of undyed cloth required in §3.4, and sew along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

## 5. Procedure

- 5.1 Agitate the composite specimen continuously for 45 min. in the soap solution at  $50 \pm 2^{\circ}$ C. (122  $\pm 4^{\circ}$ F.) at a liquor ratio of 50: 1 in the Wash Wheel (see §7.1).
- 5.2 Rinse the composite specimen twice in cold distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not exceeding 60°C. (140°F.), with the three parts in contact only along the one row of stitching.
- 5.3 Assess the change in colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.2).

#### 6. Report

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

## 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Washing: S.D.C. No. 4

NOTE—This test is identical with Washing Test No. 4 for Natural and Regenerated Cellulose in the Second Report except that assessments are now made with grey scales and not on a pass-fail basis

## 1. Purpose and Scope

1.1 The test is intended for assessing the resistance of the colour of textiles which are physically capable of withstanding the action of soap solutions near the boiling point. It is mainly intended to be used as an extremely severe test for cellulosic fibres.

## 2. Principle

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a solution containing soap and sodium carbonate, rinsed, and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

#### 3. Apparatus and Reagents

- 3.1 Wash Wheel (see §7.1)
- 3.2 Monel-metal or stainless-steel balls approx. 0.6 cm. in diameter
- 3.3 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight\*—

Free alkali calculated as Na <sub>2</sub> CO <sub>3</sub>	. 0-3% maximum
Free alkali calculated as NaOH	0-1% maximum
Combined fatty acids calculated as Na salts	85% minimum
Titre of mixed fatty acids prepared from the soap	30°C. maximum

\*Soap complying with B.S. 1912:1953 is suitable for use in this test

- 3.4 Soap solution, containing 5 g. of soap (see §3.3) and 2 g. of anhydrous sodium carbonate per litre of distilled water
- 3.5 Undyed cloths of the type under test (or, if fibre or yarn is being tested, undyed cloth made from the same kind of fibre)
- 3.6 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

## 4. Specimen

- 4.1 If the textile to be tested is fabric, sew a specimen of it, 10 cm. × 4 cm., to an equal-sized piece of the undyed cloth (see §3.5) along all four sides to form a composite specimen.
- 4.2 If the textile to be tested is yarn, knit it into fabric and treat it as in §4.1, or form a layer of parallel lengths of it between two pieces of undyed cloth and sew around the four sides to hold the yarns in place.
- 4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between two pieces of undyed cloth, and sew around the four sides to hold the fibre in place.

## 5. Procedure

- 5.1 Place the composite specimen in the container together with 10 balls as specified in §3.2 and add the necessary amount of soap and soda solution, previously heated to  $95 \pm 2^{\circ}$ C. (203  $\pm$  4°F.), to give a liquor ratio of 50:1.
- 5.2 Treat the composite specimen for 4 hr. at  $95 \pm 2^{\circ}$ c.  $(203 \pm 4^{\circ}$ F.).
- 5.3 Rinse the composite specimen twice in cold distilled water and then for 10 min. in cold running tap-water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not exceeding 60°C. (140°F.) with the two parts in contact only along the one row of stitching.
- 5.4 Assess the change in colour of the specimen and the staining of the undyed cloth with the grey scales (see §7.2).

## 6. Report

6.1 Report the numerical ratings for colour change and for staining of the undyed cloths.

#### 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

## Colour Fastness to Milling, Acid

Note— This test is identical with the test for Acid Milling in the Second Report except that assessments are now made with grey scales

#### 1. Purpose and Scope

1.1 This method is intended for assessing the resistance of the colour of wool and textiles containing wool to the action of acid as used in the milling process.

#### 2. Principle

2.1 A specimen of the textile is made into a small bag, along with specified undyed cloths, two porcelain balls are introduced, and the assembly is treated in the Wash Wheel in a solution of dilute sulphuric acid. The cloths are rinsed, separated, and dried, and the change in colour of the specimen and the staining of the undyed cloths are assessed with standard grey scales.

## 3. Apparatus and Reagents

3.1 Suitable container with means of agitation

- 3.2 Solution of sulphuric acid containing 10 g. of sulphuric acid (sp. gr. 1-84) per litre of distilled water
- 3.3 Porcelain balls 1.9 cm. in diameter
- 3.4 Two undyed cloths, each 8 cm. × 8 cm. for testing fabric and yarn or 16 cm. × 8 cm. for testing loose fibre, one piece made of the same kind of fibre as that of the textile to be tested, the second made of a fibre as indicated below or as otherwise specified—

If First Piece is-	Second Piece to be-	If First Piece is-	Second Piece to be-
Cotton	. Wool	Viscose rayon	Wool
Wool	Cotton	Acetate rayon	Wool
Silk	Cotton	Polyamide fibre	Wool or viscose rayon
Linen	Wool	Polyester fibre	Wool

3.5 Grey scales for assessing change in colour and staining of undyed cloths (see §7.2).

## 4. Specimen

4.1 If the textile to be tested is fabric, sew a specimen 16 cm. × 8 cm. on to the two pieces of undyed cloths each 8 cm. × 8 cm. (see §3.4). Sew each undyed cloth on to the coloured specimen along its four edges and in addition in one direction only at intervals of approx. 1 cm.

Weigh the composite specimen, fold to form a square with the undyed cloths inside, and sew along three outer edges. Introduce two porcelain balls (see §3.3) into the bag so formed, and sew along the fourth outer edge.

- 4.2 If the textile to be tested is yarn, knit it into fabric, and treat as in §4.1.
- 4.3 If the textile to be tested is loose fibre, comb and compress an amount at least equal in weight to the undyed wool. Lay the fibre evenly between the undyed cloths each 16 cm. × 8 cm., and sew the layers together by stitching in one direction at intervals of approx. 1 cm. Weigh the specimen, fold to form a square with the undyed wool outside, and treat as in §4.1.

## 5. Procedure

- 5.1 Place the composite specimen in the metal pot of the Wash Wheel (see §7.1) with five times its weight of sulphuric acid solution (see §3.2). Run the Wash Wheel for 60 min. at 60°C. (140°F.).
- 5.2 Remove the pattern, rinse thoroughly in running water, separate the fabrics, and dry in air at a temperature not exceeding 60°C. (140°F.). Assess the change in the colour of the specimen and the staining of the undyed cloths with the grey scales (see §7.2).

#### 6. Report

6.1 Report numerical ratings for colour change and for staining of each kind of undyed fibre tested.

## 7. Notes

- 7.1 The Wash Wheel sponsored by the Society of Dyers and Colourists can be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester 1.
- 7.2 See the following sections-
  - (a) Colour Fastness of Textiles: General Principles of Testing
  - (b) Colour Fastness: Grey Scale for assessing Change in Colour
  - (c) Colour Fastness: Grey Scale for assessing Staining.

#### COMMUNICATION

# Some Aspects of the Mechanical Finishing of Cotton Textiles

G. M. NABAB and G. P. TAWDE

Some aspects of mechanical finishing processes for cotton textiles, mainly calendering, have been examined, and the influences of heat, pressure, moisture content, and starch content on the resultant finish investigated.

An increase in moisture content of the fabric during calendering produces the following effects on the finished samples—increase in stiffness and lustre, decrease in thickness, loss in textile qualities, and increase in resistance to abrasion. On storage, both the stiffness and the lustre of the calendered fabric show a gradual fall. With softening agents, the stage at which they are added to the starch paste determines the viscosity of the paste and the stiffness of the fabric. Studies on soluble starches show that for pastes of the same viscosity the stiffness of the fabric depends on the amount of the starch taken up and on the concentration of the starch.

## Introduction

Finishing processes in general can be divided into two classes—(a) physical and (b) chemical. Among physical finishes are included the so called mechanical finishes, in which the combined effects of external agencies such as heat, pressure, and moisture are used to improve the handle of a fabric.

Although more attention has recently been paid to durable finishes, which depend on the assistance of various organic compounds, mechanical finishing still remains the main concern of a practical finisher. The modern finishes may call for specialised equipment, use of expensive chemicals, and expert technical supervision; whereas, although the equipment in mechanical finishing has often a high capital value, running costs are generally very low, the processes do not require exacting conditions, and the principles behind the application of the various finishing agents are very simple. In a recent paper, Turner and Whewell have rightly emphasised the importance of mechanical finishing in textile processing.

The effects of various mechanical finishing operations on the ultimate handle of the finished fabric are important from the customer's as well as from the manufacturer's point of view. The quantitative assessment of "handle" or "feel" is not simple, for there are various factors which affect a judgment based primarily on the sense of touch. It has, however, been shown recently that stiffness plays an important rôle in determining the handle \$3, and this property is used in evaluating the finish in the present investigation.

The present investigation deals primarily with the influence of the various working factors in calendering upon the final handle of a starched fabric. The effects have been studied of conditioning the fabric over a wide range of moisture contents before mechanical finishing. The softening brought about by lubricants has been dealt with in great detail. The connection between the viscosity of an applied starch paste and the final lustre and stiffness of the fabric has also been studied.

## Experimental

#### MATERIALS

FABRIC—For mechanical finishes: mercerised and bleached poplin, 44s warp and weft, 144 ends per inch and 76 picks per inch.

STARCH PASTE—All pastes were prepared from a selected maize starch.

For each 100 g. paste, 5 g. air-dry starch was stirred with 20 ml. cold water, made up to volume also with cold water, and heated with stirring for 45 min. in a vigorously boiling water-bath. The final volume was adjusted by adding boiling water just before completion. The paste was then cooled to 30°c. in 20 min., as it had been shown that the final viscosity depends upon time of stirring. The chief variation among different preparations was in the viscosity, and only those with values within specified limits were accepted for further use.

Application of the starch paste to the fabric was by experimental two-bowl padding mangle. Only one nip was given. The pressure of the nip was so adjusted that on passage of a control sample saturated with water at room temperature (30°c.) the take-up allowed was 80%, viz. 100 g. of air-dry cloth attained a total weight of 180 g. After padding, the fabric was dried at 50°c. for 30 min. with the samples held horizontally to avoid uneven distribution of starch through the fabric.

SOLUBLE STARCHES—These were prepared by oxidising maize starch with dilute sodium hypochlorite solution buffered at pH 8.

The viscosity-concentration relation of the starches was obtained by measuring the viscosity on a Höppler viscosimeter, which works on the falling-sphere principle.

#### CONDITIONING

The moisture content on the fabric was varied by conditioning it to equilibrium in closed chambers maintained at various humidities (Table I).

#### TABLE I4

R.H. (%)	Conditioning Agent (Saturated soln.)
50	Sodium dichromate
65	Ammonium nitrate
75	Sodium chloride
90	Glauber's salt
100	Water

## CALENDERING

The starched samples were calendered on a three-bowl Mather & Platt general-purpose

calender\*. Three types of calendering were studied, nips between a steel bowl and cotton bowls being utilised-

- (i) Swissing cold and hot (hot bowl 120°c.)
- (ii) Frictioning hot (hot bowl 120°C.)
- Chasing hot (hot bowl 120°c.).

The pressure was maintained constant throughout the work. The speed of calendering was 30 yd. per min. for ordinary calendering and 20 yd. per min. during friction calendering.

In swissing, the fabric was given a single nip, reversed face to face, and given a second nip. In chasing experiments two, three, or four layers were present in the nip at the same time.

## PROPERTIES OF THE FINISHED FABRIC

## Stiffness

Various methods have been described for measuring the stiffness of a finished fabric 2,3,5,6. In the present work, the stiffness was determined on a Gurley R.D. stiffness tester. The numerical values obtained with this instrument are directly proportional to the torsional rigidity as determined by the Peirce heart loop test 5. The instrument measures the resistance to bending of standardsize samples both with and against the curl. The resistance to bending is taken as a measure of the stiffness of the fabric. In the determination a prepared sample is clamped in a movable arm, and drawn over the top of a weighed pointer until the bending of the sample releases the pointer. The clamp arm is rotated by a wire belt running from a capstan handle, which is turned in order to move the sample. A right and left sine scale is mounted on the base. The clamp arm is rotated steadily and at a moderate rate. The stiffness of the material forces the pointer out of vertical until the end clears, or is about to clear, the vane. This is the maximum scale and is noted. The scale of deflection of the pointer is a measure of the stiffness. From the stiffness obtained in the warp and west directions, the average stiffness of the sample is taken as the square root of the product of the two 5.

All determinations were made after conditioning the fabric to 75% R.H.

#### Cuprammonium Fluidity

Damage by heat during calendering was assessed by measuring cuprammonium fluidity. starched fabrics the starch was first removed by treating the sample for 1 hr. in a 5% solution of Polyzyme (S) at 55°c., rinsing, and finally boiling with distilled water for 1 hr. until no reaction took place on spotting with dilute potassium iodide-iodine solution.

#### Thickness

This was determined by means of a compressometer at a pressure of 1 lb./sq.in. through flat circular contact plates (diameter 1 in.). The measurements were made at 65% R.H. and 26°c.

#### Lustre

An instrument which compared diffuse with specularly reflected light was employed. The sample (3 in. × 3 in.) in a fixed position was illuminated at 45° with a parallel beam of light. The source was a 100-watt tungsten-filament lamp, in a suitable lamp-house with reflector, and fed from a constant-voltage regulator.

Light reflected from the sample was made to impinge on a suitably mounted Weston Photronic boundary-layer cell. The position of the cell may be varied to measure the intensity of the reflected light at 0° and 45° to the normal. The voltage of the cell was determined by a nullpoint method in which an opposing e.m.f. was varied until no current flowed in the circuit. The reading of the potentiometer in millivolts was a measure of the intensity of the reflected light. The cell was calibrated against a standard magnesium oxide surface 7,8.

Lustre number = 
$$\frac{S_i - D_r}{S_i^{\circ} - D_r^{\circ}}$$

 $(S_i = intensity of reflected light measured at the$ 'specular" angle i (45° to the normal);  $D_r =$ intensity of the reflected light measured at the "diffuse" viewing angle r (0° to the normal); and  $S_i^{\circ}$  and  $D_r^{\circ}$  are the corresponding values for a standard magnesium oxide surface.

## Abrasion Resistance

Samples of the starched fabric after conditioning were, respectively, (a) swissed cold, (b) swissed hot, (c) frictioned. They were then conditioned at 75% R.H. and abraded on a Martindale abrasion tester of (on a 5 in. × 5 in. sample), using a load of 71 oz., against 00-grade emery cloth for 50 rubs. After abrasion, the samples were again conditioned at 75% R.H., and the resistance to abrasion was evaluated by finding the bursting strength on a Mullen tester. This was compared with the bursting strength of an unabraded sample.

#### **Experimental Results**

The results of the present investigation are divided into three parts. Part I deals with-

- (a) The effect of moisture, in the fabric at the time of calendering, on its stiffness, lustre, chemical degradation, change in dimensions, and resistance to abrasion after different calendering treatments.
- (b) The effect of pressure, temperature, and moisture during calendering on stiffness.
- (c) The effect of the variety of weave of a fabric on its starch take-up and stiffness.
- (d) The fall in stiffness and lustre of calendered fabries during storage.
  - (e) Stiffness produced by different starches.
  - (f) Stiffness with different kinds of calendaring.

In Part II a detailed study with various commercial softening agents is made, viz. Sapamine KW (Ciba), Cirrasol XL (ICI), Calsolene Oil HS (ICI), coconut oil, soap, and glycerol.

Part III deals with the results obtained with different soluble starches. The following properties are investigated-relation between viscosity and

<sup>\*</sup> The particulars of the calender are

<sup>\*</sup> The paraconne with Middle Architecture and the Architecture Architec

concentration, penetration, and the resultant stiffness.

#### PART I

The results of these investigations are shown graphically in Fig. 1-8.

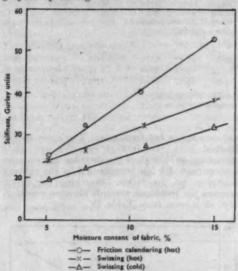


Fig. 1- Effect of Variation of Moisture Content on the Stiffness of

Fig. 1 shows the effect of the moisture content in the fabric at the time of its calendering on the final stiffness. The following observations are made—(a) There is a linear relation between stiffness and moisture content of the fabric. (b) Maximum stiffness is obtained with friction calendering; swissing through one nip with a hot bowl comes next; and swissing cold, last of all.

The influence of initial moisture content upon the final lustre of calendered samples is shown in Fig. 2. It will be seen that the maximum lustre is produced by friction calendering, swissing hot and swissing cold coming next in order. Increase in moisture content has a similar effect on lustre to that on the stiffness of the calendered samples.

The effects of moisture in the fabric during calendering on the resultant tensile strength and cuprammonium fluidity are given in Table II.

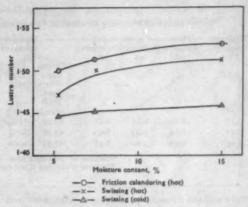


Fig. 2— Riffect of Variation of Moisture Content on the Lastre of the Fabric

Study of the table reveals that, on calendering, both starched and unstarched fabrics show similar behaviour in respect of change in tensile strength and fluidity. The rise in fluidity is negligible with the starched fabric as compared with the unstarched one, and this suggests that starch protects the cotton from both chemical and mechanical damage. The loss in tensile strength and the increase in fluidity are more pronounced with samples containing more moisture at the time of calendering. This observation lends further support to work of Wiegerink 10, who observed the same relation during a study of the effect of drying conditions on the properties of textile yarns. He has shown that the "quality index" of cellulose fibres decreases either as the temperature is increased or as the moisture content of the surrounding atmosphere is increased. Here, both breaking strength and fluidity appear to be functions of the relative humidity at which the samples are exposed.

Table III illustrates the changes in dimensions of the fabric, caused by calendering in different ways, in relation to its initial moisture content. To study the relative change in dimensions along warp and weft a square was marked on the fabric. The fabric after conditioning for three different moisture contents (as given in the first column) was calendered by swissing hot with one nip, and

TABLE II

Effect of Conditioning the Fabric on its Tensile Strength and Fluidity during Calendering

Mode of Calendering	Moisture Content of	Tensile (lt	Strength	Fluidity (reciprocal poises)		
	Fabric (%)	Unstarched	Starched	Unstarched	Starched	
Not calendered	7-4	22-0	22-9	13-5	13-5	
Swissing, cold	7-4	_	September 1	13.5	13-5	
	15-1	_		13-8	13-4	
Swissing, hot	7-4	21-7	22-0	14-8	14-1	
	16-1	20-9	21.7	15-0	14-8	
Frictioning, hot	7-4	21-3	22-2	15-4	14-6	
AT LOT AND A STATE OF	15-1	20-6	21-4	16-8	14-8	

TABLE III

Effect of Moisture	Content in	Conditioning o	n Dimensions	of Calen	dered Fabric
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Moisture Content	Thickness* (0-001 in.) after Swissing Swissing Frictioning			Dimensions of Marked Area (mean of 10 measurements)				Change produced (%) Length Length Area			
(%)	. cold	hot	hot	Longth	Area	Length	Final Length	Area	Warp	Weft	
				Warp and Weft	(sq. om.)	Warp (em.)	Weft (cm.)	(aq. cm.)			
				(cm.)							
5-17	7.35	6-16	5-87	81-21	6595	80-35	81-87	6578	-1.06	0.81	-0.26
7-41	7-18	6.03	5.65	81.21	6594	80-22	82-24	6598	-1.21	1.27	Nil
15.00	6-15	5-80	5-47	81-21	6595	79-81	83-27	6646	-1.72	2.54	0.77

\*Thickness of unstarched fabric (not calendered) 0-00742 in.
Thickness of starched fabric (not calendered) 0-00743 in.

the resultant dimensions of the area were measured by means of a micrometer microscope. From Table III it seems that, for a particular mode of calendering, the thickness is a minimum when the fabric has the maximum moisture content. The results for the thickness are depicted graphically in Fig. 3, which shows that the effects produced by frictioning and by swissing hot are similar. During cold calendering, as there is no applied heat, the decrease in thickness becomes apparent only after increasing the moisture content of the fabric beyond 8%.

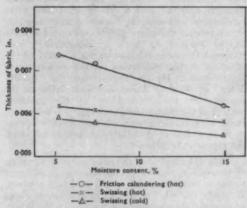


Fig. 3— Effect of Variation of Moisture Content on the Thickness of the Fabric

The fabric would be expected to become more plastic with increase in temperature, and this is confirmed by measurements, first of the decrease of thickness and then of the increase of area, after passage through one nip at standard pressure. Both of these quantities increase with increased working temperature. Similarly, plasticity is shown to increase with moisture content, since the increase in area caused by one passage is greater as the moisture content is higher.

The results of measurements of abrasion resistance of starched fabric are set out in Fig. 4. They show that moisture plays an important rôle in increasing this resistance to abrasion. The increase is most marked after swissing with a hotbowl calender as compared with that from either

cold swissing or hot frictioning. The lower results obtained with the friction calender are quite interesting, as they show the decrease in resistance to abrasion which has been caused by the rubbing undergone by the fabric. Starching the fabric increases the resistance towards abrasion considerably, as is seen from Table IV.

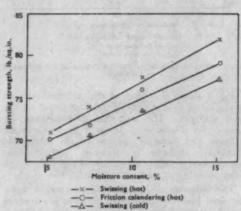


Fig. 4— Effect of Variation of Moisture Content on the Resistance to Abragion of the Fabric

TABLE IV

## Effect of Starching on Resistance to Abrasion

	Before Abrasion After Abra			
Unstarched fabric (not calendered)	84-0	67-2		
Starched fabric (not calendered)	85-8	74-1		

It was observed during preliminary experiments on calendering that the temperature of the hard calender bowl and the pressure applied to it play an important part in determining the final stiffness of the finished fabric. In order to study this effect in detail, a test was carried out with starched fabric. As it was not possible to record the pressure applied on an ordinary calender, the experiments were carried out using a Schreiner calender, on which both the temperature and pressure can be easily varied and recorded. It will be seen from Fig. 5 that the stiffness increases fairly rapidly up to

90°c. and then more gradually. For the range of pressure studied, the stiffness increases linearly with the applied pressure.

Study of Fig. 6 reveals some points of special interest. Three fabrics of different weaves (poplin, twill, and sateen), made from the same yarn, were starched and calendered. It was observed that they absorbed varying amounts of starch during padding. The straight-line relation between starch uptake and increase in stiffness shows that the latter is influenced mainly by the amount of starch in the fabric and not by the nature of the weave.

It is well known that calendered goods tend to lose their lustre on storage. However, no data are available about the actual change. The samples, after calendering, were stored at 75% R.H., and the stiffness and the lustre were measured from time to time. From Fig. 7 and 8 it seems that the greatest fall in stiffness takes place during the first month of storage. With lustre, the fall is more gradual. Both lustre and stiffness fall most rapidly in friction finishes, less after hot swissing, and least after cold swissing.

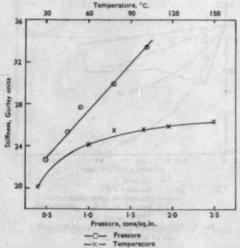


Fig. 5— Variation of Stiffness with Temperature and Pressure of the Calender Rowl

Each type of starch possesses distinctive properties, which are utilised in obtaining different effects in finishing. The data for stiffness produced by different starches at two different relative humidities of conditioning for hot-bowl and friction calendering are embodied in Table V.

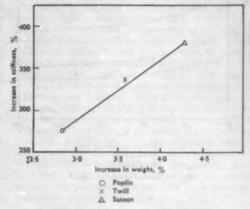


FIG. 6- Effect of Weave on Starch Take-up and Increase in Stiffness

From the results it will be seen that, in addition to the viscosity, other factors, such as swelling, penetration, etc., are also important in determining the stiffness of the fabric. The increase in stiffness produced by increased moisture content is remarkable for arrowroot. The potato starch paste, although of low viscosity, gives stiffness which is comparable with that produced by rice, maize, and wheat starches. Rice is known to give a stiffer feel than most other starches.

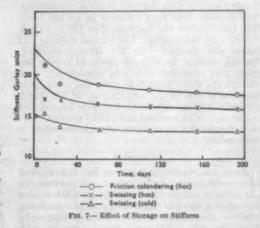


TABLE V

## Viscosity of Paste and Stiffness of Finish for Different Starches

		HITCH THE SER								
Starch				Viscosity of						
		Ho Swiss			(Gurley units) Hot Friction			2% Paste		
		75% R.H.	100% R.H.		75% R.H.	100% R.H.		(centipoises)		
Arrowroot		28-8	46-5		39-6	63-8		6-53		
Sago	490	23-3	39-4		32-3	52-8		4-88		
Potato		31-4	45-6	,	39-8	53-6		3-32		
Rice		36-2	53-2		41.3	60-6		7-85		
Maize	***	33-2	42-9		37-0	66-0		8-74		
Wheat	800	32-0	43-7		38-1	62-2		8-03		

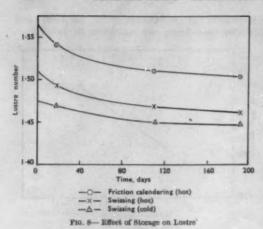


TABLE VI

## Effect of Mode of Calendering on Stiffness and Lustre

Mode of Calendering		Stiffness (Gurley units)		Lustre Number
Swissing cold	***	22-8		1.45
Swissing hot	***	26-8		1.50
Frictioning hot		32-2		1.52
Chasing-				
lat passage		14-5	1	
2nd passage		13-4	>	1.48
3rd passage	***	13.3	)	

Table VI gives the comparative stiffness and lustre resulting from different types of calendering. The pressure was kept constant in all the tests. The temperature was maintained at 120°c., except during cold calendering. The highest stiffness and lustre are obtained with the friction calender. During chasing, the stiffness produced is even less than that obtained with cold calendering, even though the metal bowl is heated. The decrease in stiffness is due to the breaking up of the starch film when two layers of the fabric come into contact in the nip. With a second and a third passage, there is a further fall in stiffness, though the effect is very small.

#### Discussion

From the results discussed so far, it is evident that moisture plays an important part in increasing the stiffness and the lustre of a starched fabric after calendering by all of the three modes of calendering studied, the maximum effect being produced by frictioning hot, and the minimum by swissing cold. This is taken to be related to the increased plasticity of the cloth and the starch, through the combined action of heat and moisture. The increase in lustre with increased moisture content seems to be due to the greater ease of flattening and polishing the surface.

The greater decrease in thickness and increase in area with a fabric containing a higher moisture content at the time of its calendering are obviously accounted for by the increase in plasticity of the fabric.

The increase in resistance towards abrasion of the calendered cloth may also be referred to the same cause, since the higher the plasticity the more compact the structure.

The increase in cuprommonium fluidity and the decrease in tensile strength with a calendered fabric containing higher moisture content prior to calendering may be due to the increase in reactivity conferred by swelling of the fibres.

## PART II

The softening effects brought about by some of the commoner softening agents used in conjunction with starch paste are seen from Fig. 9. With Sapamine KW and Cirrasol XL, the effects of their addition before and after the gelatinisation of starch were studied. For the former, the softening agent was added at the start of boiling; for the latter, after heating for 30 min. The viscosity (at 30°c.) of the resultant paste and the moisture content of the fabric after starching and conditioning at 75% R.H. were determined.

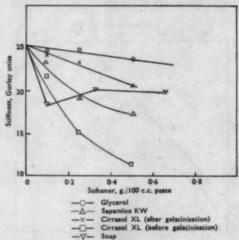


Fig. 9- Softening produced with Various Softeners

These results showed that surface-active softening agents bring about softening by lowering the viscosity of the starch paste. They also cause a decrease in moisture content in the fabric with resultant low plasticity. With Cirrasol XL and Sapamine KW it is observed that maximum softening is brought about when they are added before the gelatinisation. When they are added after gelatinisation there is some softening, but the total effect is small. In low concentration, soaps behave predominantly as mild alkalis, and lower the viscosity; but as the soap concentration is increased the viscosity, after reaching a minimum, rises rapidly. This effect is not due to increase in alkalinity alone, but must be ascribed to chemical reaction or association between soap and starch.

The observation of a lowering in moisture content in the fabric treated with glycerol lends further support to an observation of McPherson <sup>11</sup>, during his work on equilibrium moisture content at various relative humidities of cotton linters treated with various amounts of glycerol. He showed that, at all humidities, the glycerol-cellulose system contained less water than would the glycerol and cellulose separately.

#### PART III

The viscosity-concentration relationships were obtained for four different starches, and from them the concentrations of the starches required to give a paste viscosity of 10 centipoises were determined. These values are given in column B of Table VII.

#### TABLE VII

#### Effect of Concentration of Starch on Stiffness and Increase in Weight of Fabric

Type of Starch	Conen. of Starch to give Viscosity of 10 centipoises (g./100 c.c.)	Stiffness of Starched Fabric (Gurley units)	Increase in Wt. of Fabric (%)
A	В	C	D
-1	2.05	26.0	1.1
2	6-10	26-9	2-6
3	20.75	34-2	14-4
4	34-50	53-6	27.0

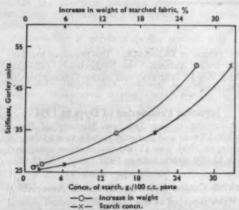


Fig. 10— Relationship of Stiffness to Concentration of Starch and its Take-up by the Fabric

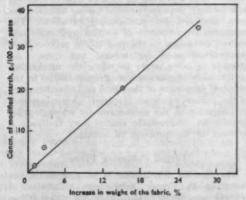


Fig. 11—Relationship between Concentration of Starch Pasts and Increase in Weight of the Starched Fabric

The stiffness of the fabric after padding in these pastes and then calendering with a hot bowl is given in column C. The results in column D were obtained by weighing the fabric before and after padding (after conditioning at 75% R.H.). From Fig. 10 it is seen that for the series of starches the relation between stiffness and increase in weight follows a similar course to the relation between stiffness and the concentration required to give a paste of the standard viscosity. By cutting sections of calendered specimens and staining with dilute iodine solution, it was shown that the stiffness increases as the starch penetrates further into the fabric. Thus, penetration, as determined by increase in weight of the fabric, is an important factor in deciding its stiffness, which, in turn, is dependent on the extent of degradation of the starch. This is seen from the straight-line graph obtained when increase in weight is plotted against the concentration required for a paste of a particular viscosity (Fig. 11).

#### LUSTRE

The term lustre is applied, in its most general sense, to denote the property of a body whereby a large proportion of an incident beam of light is regularly reflected and only a small proportion is scattered. In textiles and some other objects there is a further distinction, bound up with the characteristic surface texture, between gloss and lustre. Gloss implies reflection from a surface which approaches true planarity, so that the incident beam is reflected without subdivision at the angle of reflection, and the observer, according to his position, may see either large unmodified bright or dark areas on the surface, "Lustre" in textiles is generally understood to mean reflection from a large number of small surfaces, all approximately parallel, so that total reflection is still largely specular, but a single large incident beam is reflected as a great number of parallel small ones. The play of highlights and shadows still persists as the relative positions of observer and surface change, but the differentiation is less sharp, and highlights are softer.

In assessing lustre on paper or calendered plain cloths, in which the small reflecting surfaces are all set parallel to the overall surface of the object, it has been customary to direct an incident light beam at 45° to the normal to the surface and then, by means of a suitable photometer head, to measure the intensity of the reflected light in the direction of (a) the normal and (b) 45° on the opposite side of the normal. Measurement (a) will then indicate the proportion of light which is diffusely reflected, and (b) that which is specularly reflected. The ratio (b)/(a) then measures the efficiency of directed reflection, the "gloss" or "lustre" according to the kind of surface. With a perfect mirror it would be infinity, with a perfect matt surface it would approach unity.

This was the method adopted in the present work, and lustre measurements to show the effect of calender treatments are given. Further consideration has shown that they are directly applicable to swissing finishes, but not so suitable

for Schreiner finishes. In the former a normal to the surface of the cloth is normal to most of the constituent small reflecting surfaces. The interpretation given is satisfactory, and rise in the ratio truly signifies increase of lustre.

DEPARTMENT OF CHEMICAL TECHNOLOGY UNIVERSITY OF BOMBAY Вомвач 19 INDIA

(Received 18th October 1954)

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## CORRESPONDENCE

The Editor dose not hold himself responsible for opinions expressed by correspondents

## Colour Changes of Dyes

A number of our graduate students are investigating the physical structure of dyes when their colour change is influenced by temperature.

They would appreciate the help of your members by any communication to me, giving the name, number, and manufacturer of any dye in an ionic or non-ionic solution that undergoes a colour change at any temperature below 10°c.

ROBERT S. FINN

MATHEMATICS DEPARTMENT University of Southern California 3518 University Avenue Los Angeles 7 U.S.A.

28th February 1955

## Notes

## Meetings of Council and Committees May

Council-18th

Finance and General Purposes-5th and 18th

Publications-17th

Colour Index Editorial Panel-12th

Diplomas - 25th

Diplomas Executive Subcommittee-2nd and

Light Fastness Subcommittee- 10th

Perkin Centenary-20th

Mercer Lecture-20th

## Fees to be paid by Applicants for the Fellowship

Applicants for the Fellowship are reminded that their application must be accompanied by a fee of £3 3s. Od. A further fee of £7 7s. Od. must be paid by successful applicants before their names can be entered on the register of Fellows.

#### Confederation of Textile Dyers and Finishers

Mr. Philip N. Hoyle has been elected Chairman in succession to Mr. W. Crossley.

## President of the Textile Institute

Mr. W. T. Winterbottom, C.B.E., has been elected President of the Institute in succession to Mr. C. H. Colton.

British Exports of Dyes

Exports of dyes from the United Kingdom were about 30% higher in 1954 than in 1953 (262,819 cwt. valued at £10,697,816 compared with 209,243 cwt. valued at £8,356,919). The principal markets were India, Australia, the Netherlands, Pakistan, Hong Kong, Belgium, and other Commonwealth countries.

## Japanese Production of Dyes in 1954

According to the Japanese Ministry of International Trade and Industry, production of dyes in Japan in 1954 was 17,000 metric tons compared with 19,000 metric tons in 1953.

## British Cotton Industry Research Association

Work reported at the thirty-fifth annual general meeting included the use of an automatic size box fed with cold starch slurries. New sizes are being studied from the point of view of ease of removal in finishing. An experimental raising machine has been designed for fundamental studies. minimum requirements of sodium hydroxide and sodium dithionite in the pad-steam process have been investigated for several vat dyes. An attempt is being made to relate the mechanical performance of crease-resisted rayon fabrics to the physical properties of the fibres and the chemistry of the various processes involved. A fundamental investigation of the mechanism by which textiles acquire an electrostatic charge may facilitate a solution of the problem of soiling.

## Tergal Polyester Fibre

The Société Rhodiacéta is at present manufacturing in France pilot-plant quantities of polyethylene terephthalate fibre, for which it has adopted the brand name Tergal.

## New Books and Publications

## Praktikum der gesamten Kleiderfärberei

By C. Blau and C. F. Strubel. Pp. 512. Munich: Franz Eder Verlag. 1954. Price, DM 20.00.

In reviewing a book of this type, the reviewer must of necessity consider its contents in the light of his own experience in the field which the book covers. This review is, therefore, intended to evaluate the book from the standpoint of the British garment dyer.

The first section of the book is devoted to a miscellany of supplementary topics, such as dyeworks layout, boilers, boiler waters and treatment, materials of construction, and so on. Whilst providing a background of interest, it is difficult to see the value of this section. The material therein is insufficient to be of practical value, and the majority of the section could be omitted without adverse effect on the book.

The second section deals with the theoretical background of dyeing adequately. It is in effect an elementary textbook on dyeing principles and theory, providing a background sufficient to enable the practical garment dyer to form an intelligent appreciation of the processes which he

The practice of garment dyeing is dealt with in Section III. It is at this point that the book gives a picture of a trade which differs considerably from the British trade. The author deals with a variety of subjects under this heading, including, for example, loose wool dyeing, batik dyeing, and hand printing, techniques which do not come within the garment dyer's field in this country. Much space is devoted to the application of vat, sulphur, and azoic dyes to a variety of fibres and to unions. Such methods, whilst of technical interest, and of use in occasional special cases, are not economically practicable in our own

With some omissions and with some condensation this could be a useful textbook for the apprentice garment dyer. The experienced garment dyer will find little that is new to him.

The literature of garment dyeing is so sparse that an authoritative textbook on the subject would be very welcome. It must be recorded with regret that this book does not quite fill this gap.

C. BELLHOUSE

## Silicones and their Uses

By R. R. McGregor. Pp. xv + 302. London: McGraw-Hill Publishing Co. Ltd. 42s. 6d.

Both the chemistry and the application of silicones continue to grow at a steady rate, and many textile technologists have been hard pressed to keep abreast of developments. This book brings together much scattered material that has appeared over the last few years, and will greatly help readers to correlate the different types of silicone products both chemical and commercial.

The early history of organosilicon chemistry is fascinatingly described in the first section, followed by a useful chapter on commercial development of silicones. Commercial silicone products are divided into five classes-fluids, compounds (silicone fluids plus finely divided silica), lubricants (silicone fluids plus soaps), resins, and rubbers. The main textile interest, which is in the use of silicones as water-repellents, is only very briefly described. Another chapter gives lists of industries with their known silicone applications. Classified under the textile industry are uses of silicones for coating fabric or conveyor belts; as defoamers; as electrical insulators for spinner bucket or dyehouse motors; as lubricants for sewing threads, tenter frames, or drying cans; or as non-stick agents for textilecoating equipment. Although this is useful, it detracts from the readability which otherwise is a feature of the book. A final section deals clearly with the more detailed chemistry of silicone preparation.

The author has had access to authoritative industrial sources in compiling the work, and the advantage of advice from notable workers in this field. The book, which is well indexed and beautifully printed, may be recommended with confidence to those requiring an introduction or overall survey of silicone chemistry and G. LANDELLS applications.

# The Microscopy of Animal Textile Fibres including Methods for the Complete Analysis of Fibre Blends

By A. B. Wildman. Pp. viii + 209. Illustrated with 88 line drawings + 235 half-tone + 11 colour photomicrographs. Leeds: Industries Research Association, 1954. Price. 42s. 0d. (21s. 0d. to members of W.I.R.A.).

The practical importance of reliable methods for identifying animal fibres and estimating their proportions in mixtures has been recognised for many years in the U.S.A., where the Wool Products Labeling Act of 1939 specifies that, if a fabric is sold as containing a certain animal fibre, the quantity of that fibre present must also be indicated. In this country the subject is obviously of direct interest to manufacturers who export to the U.S.A., while similar legislation in other countries, including the United Kingdom, is contemplated. The limitations of physical and chemical tests render the microscope indispensable for fibre identification. Unfortunately, however, existing publications on the microscopy of animal fibres do not cover a sufficiently comprehensive field in sufficient detail. It was with the aim of remedying this defect that the present work was published. The author, who is Chief Biologist of the W.I.R.A., has wide experience in this field, and has at his disposal an extensive range of authentic fibre samples which has been examined with modern photomicrographic equipment. The book is divided into five parts, as followsPart I, on comparative morphology, gives an outline of the growth and the structure of animal fibres, as illustrated by the author's work on the coat and fibre development of some British sheep. A brief account is also given of the fundamental structure of cellulosic fibres used for textile purposes.

Part II deals with sampling and preparation of fibres for identification and for the qualitative analysis of mixtures. Working details are given for the proportion of whole mounts and crosssections, as well as for the examination of scale structure. The advantages and the disadvantages of various mounting media are given, and their suitability for various purposes is outlined. Preparation of cross-sections by means of the Hardy, Schwarz, or Mennerich microtomes is described, and this is followed by an account of how to make preparations which demonstrate the scale pattern. Scale examination is divided into two categories-study of surface casts and of rolled impressions. The latter technique, which was developed in the author's laboratory, gives a record of the entire surface scale pattern of a fibre, and constitutes a most valuable advance in fibre microscopy, since casts give an indication of the condition of only a portion of the fibre surface, and may in certain instances be misleading. The final chapter of Part II, written by one of the author's colleagues, gives an account of the photomicrographic procedure employed in this study of animal fibres.

Part III, which extends to 125 pages, commences with the classification of medullae and cuticular-scale patterns. The system of nomenclature put forward constitutes a definite advance, and it is to be hoped that it will be quickly and widely adopted. In the remaining eight chapters (some 115 pages) detailed descriptions are given of the structure of animal fibres of industrial significance, the letterpress being well illustrated with photomicrographs. Information on the origin and the uses of the fibres is given, and although this does not strictly fall within the scope of fibre microscopy, it presents a background of definite use in the examination of commercial material.

Part IV deals with quantitative analysis of fibre mixtures. The methods of sampling and analysis are described, and data given on the number of counts and measurements required to give results of required maximum errors.

In Part V the author gives an account of present views on the fine structure of animal fibres based on microscopic examination, supplemented by the use of electron, phase-contrast, and ultraviolet microscopy.

The book is clearly printed on good-quality paper and gives 104 references to the literature. Photomicrographs and drawings are excellent. These features, together with the manipulative details given, make the book one which can be thoroughly recommended to all interested in the microscopy of animal textile fibres.

J. L. STOVES

# Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

## I-PLANT: MACHINERY: BUILDINGS

New Finishing Machinery. H. F. Creegan. Amer. Dyestuff Rep., 44, P136-P138 (28 Feb. 1955). A survey of new finishing machinery deals in special

A survey of new finishing machinery deals in special detail with the Barotor piece-goods pressure dyer, the Vapojet Dryer employing superheated steam, and the Eicken-system instruments for measuring moisture, which enable practical automatic control to be attained in dyeing fibres, yarns, or fabric.

J. W. B.

#### PATENTS

Safety Systems for Carbon Black Plants. Phillips
Petroleum Co.

A system for preventing explosions in burning of
material during the manufacture of carbon black.

C. O. C.

Aftertreating Degummed Stalk Fibres. J. E. Cary, R. E. Shafer and V. Cary. BP 726,111

A machine in which all processes subsequent to decortication, i.e. degumming, washing, and bleaching, of stalk-type fibres can be performed, and which can alternately hold opposite ends of fibre bundles and treat the free end, is described. The conveyor consists of a relatively wide and elongated rubber belt with an undulated upper surface, against which press rubber-tyred wheels with oircumferences concentric with the arcuste undulations; similarly opposed paddles ensure adequate circulation and pulsation of liquid. J. W. B. Washing Freshly-formed Artificial Threads. Textile & Chemical Research Co. BP 726,710
Apparatus in which the threads are readily washed

while the tension in them is being controlled prior to winding.

C. O. C.

Thread-advancing Reels. Courtaulds.

BP 726,887

Dyeing Machine. T. R. Purves. BP 727,430 More even flow of liquor and better penetration are obtained in a dyeing machine in which the liquor is circulated by means of a propeller placed in an auxiliary chamber alongside the dyeing vessel if this chamber is connected to the top and the bottom of the main vessel by two lateral tapered passages, the narrowest cross sections of which are adjacent to the main vessel. C. O. C.

Coating Textiles with Flock. Velveray Corpn. USP 2,675,330

Machine for applying flock to a fabric coated with adhesive as it moves through a unidirectional field supplied from an A.C. source and which provides means for preventing a breaking or "treeing" of the flock as it approaches the adhesive surface.

C. O. C.

Web Coating Apparatus. J. Waldron Corpn. BP 727,602
An air nozzle for use on a web coating machine has an air jet in slot-form, defined by a pair of spaced converging plates, and a buffle plate set at an acute angle to the outer surface of one of the plates to form a discharge slot adjacent to the air jet so that the high-velocity air flow through the jet will induce secondary air flow through the jet will induce secondary air flow through the discharge slot. The position of the baffle plate is adjustable.

Both air jet plates may have separate lip portions, one being deflectable; the width of the slot may be adjustable, and a locking device is provided to maintain any required position of the rotatable nozzle which is mounted close to a backing roll over which the web to be coated is passed. A deflector and a drip pan are located below the jet to catch excess coating material removed by the jet.

Applying Liquid to a Moving Surface. Molins Machine Co. RP 727.478

A device for regulating the quantity of liquid supplied to an applying roller comprises a regulating roller with e.g. transverse helical grooves, and raised portions between the grooves so that on rotation the raised portions touch in turn the liquid-carrying surface of the applying roller; means of supplying the liquid to the roller, e.g. from a bath, and means of altering the speed of rotation of the regulating roller to vary the quantity of liquid carried forward. The length of the grooved part of the roller may be varied by using short interchangeable sections.

8. V. S.

Platen Felting Machine. N. Wahl. BP 726,680 A felting machine has a jogger platen and a number of spindles arranged vertically to the platen. The spindles can be adjusted axially during jogging to control the pressure applied by the platen. C. O. C.

Transfer Printing Machine. May Hosiery Mills. USP. 2,674,056 An automatic machine for applying transfers to hosiery. C. O. C.

Moulding Skins and treating them with Liquids. Secotan. BP 726.828

A machine in which skins are moulded into shape, e.g. for shoe uppers, and in which they are tanned, fat liquored, dyed etc. while on the mould.

C. O. C.

Rotary Press for applying Wax Carbon to Sheets. McBee Co. USP 2,674,225

#### III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Interaction of Bivalent Metal Ions with N-Hydroxyethylethylenediaminetriacetic Acid. S. Chaberek and A. E. Martell. J. Amer. Chem. Soc., 77, 1477-1480 (20 March 1955).

Potentiometric study of the formation of 1:1 metal chelates between N-hydroxyethylethylenediaminetriacetic acid (I) and bivalent Cu, Ni, Co, Zn, Cd, Mn, Fe, and Ca ions shows that, compared with ethylenediaminetetracetic acid (II), replacement of an acetate by a hydroxyethyl group decreases the basicity of the amino acid anion and correspondingly reduces the chelate formation constants by 0-9-3-4 log K units. The affinity of I for bivalent metal ions compares favourably with that of II in the acid pH range, but the chelates of I are considerably more susceptible to metal hydrolysis and other interferences in alkaline solutions. In view of its high affinity for ferric ions in alkaline solutions and its high solubility in water at all pH values, I is a valuable addition to the range of metal-chelating agents.

C. O. C.

Dependence of the Solubilising Powers of Sodium Oleate Hydrosols on their Structures. Z. N. Markina, K. A. Pospelova, and P. A. Rebinder. Doklady Akad. Nauk S.S.S.R., 99, 121-124 (1 Nov. 1954).

Viscosity and deformation measurements are made on Na oleate soln., and the results are compared with data on the solubilising powers of these soln. for organic liquids (acrylonitrile, octyl alcohol, benzene, cyclohexane, octane, dodesane). Structural viscosity becomes detectable in Na oleate soln at a concen. of 80 g,/litre and then increases rapidly with further increase in concn., but no true gel properties develop, indicating absence of three-dimensional networks. This change in structural viscosity is accompanied by rapid increase in solubilising power. It is considered that, in the course of this change, the hydrated spheroidal micelles present at low concn. give way to asymmetrical lamellar micelles, which can form compact laminates of high internal surface that are more readily penetrated by organic liquids. A. E. S.

Chemistry of Alginic Acid. R. Köhler. Chim. et Ind., 73, 283-289 (Feb. 1955). Review with 46 references. C. J. W. H.

Action and Formulation of Antistatic Preparations.
W. Sprenkmann. Melliand Textilber., 36, 174-177 (Feb. 1955).

A short review of previously published German work in idem. An instrument for laboratory evaluation is illustrated. 9 references. 8. R. C.

Unhairing Liquors from Staple-rayon Wastes. I. Skogestad. J. Amer. Leather Chem. Assoon., 49, 757-760 (Dec. 1954).

Sequestering Agents for Polyvalent Metals, Dow Chemical Co. BP 727,482 Compounds of formula—

(R¹ = H, Alk which may be substituted by OH or alkoxy and R² = hydroxy Alk; or R² = hydroxy Alk and R³ = Alk or hydroxy or alkoxy.Alk), e.g. a monolactone of hydroxyaminemonoacetic acid, are sequestering agents for polyvalent metals particularly ferric, and chromic ions and the ions of Co, Cu, Ni and other transition elements. They are effective over a wide pH range but have little sequestering effect on alkaline earth metals.
BP 727.483

Quaternary Ammonium Salts of Formaldehyde Sulphoxylic Acid. Br. BP 727,472
The quaternary ammonium salts of formaldehyde sulphoxylic acid, e.g. (CH<sub>2</sub>)<sub>4</sub>NHSO<sub>2</sub>, HCHO, are soluble in water and a wide range of hydroxylic organic solvents but are not readily hydrolysed. Their viscous solutions in diethylene glycol, glycerol and triethanolamine are suitable for use in textile printing pastes. C. O. C.

NN'-di-(2-Chlorophenyl)-ethylenediamine— Gas
Fume Fading Inhibitor. Celanese Corpn. of
America.

NN'-di-(2-Chlorophenyl)-ethylenediamine—
BP 727,852

NN'-di-(2-Chlorophenyl)ethylene diamine is a highly effective gas fume fading inhibitor which itself has little tendency to discolour when exposed to gas fumes.

Guanamine-Formaldehyde Resins for Textile Finishing, American Cyanamid Co. BP 727,425
A guanamine-formaldehyde resin can be obtained as a stable, dry, free-flowing, cold water-soluble powder by treating 1-4 mol. HCHO with 1 mol. aceto- or propioguanamine at 80-100°C. until a resin syrup is obtained which has maximum dilutability at 25°C. with 20% aq. KCl and with water of 4 4% resin solids respectively. The pH is then adjusted to 70-9-5 and solution evaporated dried at 25-325°C. The product is used for imparting crease-resistance and/or dimensional stability to textiles, the finished goods are very little discoloured when subjected to chlorine-containing bleaches. C. O. C.

Triphenylmethane Mothproofing Agents, FBy.

BP 726,789

Mothproofing agents of formula-

(Y and Z = H, Hal, Alk, alkoxy, amino or acylamino; m and n = whole numbers) are obtained by treating appropriate of-dihydroxytriphenylmethanes in alkaline medium with di-halogenomethanes at 100°C. Thus 1 mol. of sodium 2: 2'-dihydroxy-3: 5: 3': 5': 4"-pentachlorotriphenylmethane-2"-sulphonate is dissolved in 2-2:5 mol. of dil. caustic soda and stirred for 5-10 hr. at 100°C. with 1-1:5 mol. of methylene chloride to yield the colourless

Application of 1.5% of this methylene other from a neutral or acid dyebath gives wool protection against textile pests. C. O. C.

Quaternary Phosphonium Compounds as Mothproofing Agents. Gy. Compounds of formula—

(A = halogen substituted monophenyl, diphenylyl or phenoxyphenol; Y = O or S; Z = alltylene of 1-3 C; n = 0 or 1; X = Hal) e.g. 2:4-dichlorophenoxyethoxymethyltriphenyl phosphonium chloride, are highly effective mothproofing agents, being more effective than previously known compounds of similar type and often having better wet fastness properties. C. O. C.

#### Aqueous Solution of Silk. T. C. Whitner.

USP 2,675,326
Copper hydroxide dissolves readily in an aqueous solution of an alkali metal hydroxide or of a tetra alkyli ammonium hydroxide if a water-soluble polyhydroxy-alcohol, e.g. ethylene glycol, diethylene glycol or sucrose, is present. Such solutions are suitable for dissolving silk to produce solutions suitable for impregnating cellulose

fibres with subsequent acid treatment to precipitate the silk in silu.

C. O. C.

4-Alkoxy-2-hydroxybenzophenones for stabilising Organic Polymers to Ultraviolet Radiation.

I.C.I.

BP 726,792
Incorporation of 4-alkoxy-2-hydroxybenzophenones,
e.g. 4-methoxy- or 4-ethoxy-2-hydroxy benzophenone,
protects organic polymers against deterioration by ultra
violet radiation.

C. O. C.

Epoxy Resin and Polyamide Compositions. General Mills. BP 726,570

The epoxides obtained by reacting polyhydric alcohols with compounds containing epoxy groups are soluble in ketone solvents and can be used as catalysts for curing polymeric fatty acid-aliphatic polyamine condensates to produce rubbery highly resilient and extremely flexible compositions. These compositions have wide uses, e.g. for glossing paper, as textile finishes, etc. C.O.C.

Plasticised Polyvinyl Chloride Compositions for Coating Bookbinding Cloths. DuP. BP 726,577 Polyvinylchloride or a copolymer of vinylchloride

Polyvinylchloride or a copolymer of vinylchloride plasticised with a blend of tricresyl phosphate and polya-methylstyrene when used for coating bookbinding cloths yields a surface to which drying oil inks adhere. In addition when pressed against cloths finished in other ways, e.g. with collulose nitrate, there is no migration of the plasticiser from the polyvinyl chloride coating. C.O.C.

# IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Mobility of Halogen in the Benzene Nucleus. V. N. Ufimtsev and M. M. Malafeeva. Doklady Akad. Nauk S.S.S.R., 99, 555-558 (1 Dec. 1954).

A measure of the mobility of nuclear bromine in benzene and naphthalene cpd. is obtained by determining their rates of reaction with piperidine (or o-bromoaniline for very active cpd., e.g. 1-bromo-2:4-dinitrobenzene). The effect of the position of a nitro group on the mobility of bromine increases in the order meta < para < ortho, the respective rates of reaction relative to that of bromobenzene (= 1) being, very approx., 60, 9,000, and 40,000. Addition to copper sulphate results in great increase in the mobility of the least active cpd., but has little effect on cpd. of moderate or high activity. A. E. S.

Sulphonation of Halogen-substituted Benzenes. V. O. Lukashevich. Doklady Akad. Nauk S.S.S.R., 99, 995-998 (21 Dec. 1954).

990-998 (21 Dec. 1904).

Difficulties in the sulphonation of polyhalogenobenzenes can be avoided by use of a large excess of 60-65% oleum as sulphonating agent, generally at room temp.; the product is often largely or almost entirely the sulphonic anhydride. By this method monosulphonic acids are prepared from 11 mono-, di-, tri-, and tetra-chlorobenzenes, 10 mono-, di-, tri-, and tetra-bromobenzenes, and 3 mono-and di-iodobenzenes.

Ultraviolet Absorption Spectra of some Vinylnaphthalenes and Related Aromatic Hydrocarbons, L. H. Klemm, H. Ziffer, J. W. Sprague, and W. Hodes. J. Org. Chem., 20, 190-199 (Feb. 1955).

Influence of Caffeine on the Coupling Reaction between β-Naphthol and p-Diazobenzenesulphonic Acid. J. T. G. Overbeek, C. L. J. Vink, and H. Deenstra. Rec: Trav. chim., 74, 69-76 (Jan. 1955).

The rate of coupling between  $\beta$ -naphthol and p-diazobenzenesulphonic acid at pH 5-09 is lowered by the presence of caffeine. This effect can be explained by 1:1 complex formation between caffeine and the two reactants, for which there is independent evidence from solubility measurements. Complex-formation constants are given. Rate constants at 20°C. are determined, and the reaction is shown to be bimolecular. The results suggest that the reaction is between (i) free  $\beta$ -naphthol and free diazo compound, or (iii) free  $\beta$ -naphthol and complex diazo compound. Reaction between complex  $\beta$ -naphthol and complex diazo compound is unlikely. A. J.

Kinetics of a Polymorphic Transformation of Azoxybenzene. A. Hodkin and D. Taylor. J.C.S., 489-497 (Feb. 1955).

A new monotropic polymorph of azoxybenzene is characterised, and the kinetics of its transformation into the stable form under thin-film conditions is investigated from 0° to 30°c. by a photometric method. The results lead to the same mechanism as that proposed by Hartshorne for the monoclinic S → rhombic S transformation, viz., one based on the difference in rates of escape of molecules from the two adjacent lattices, and requiring the full latent heat of sublimation of the metastable form as energy of activation, viz. 23±1 kcal./mole. An approx. doubling of the rate after exposure to ultra-violet radiation is reported and discussed.

Inhibiting Effect of Trypan Blue on the Experimental Production of Liver Cancer. S. Iwase and K. Fujita, Nature, 175, 552 (26 March 1955).

Trypan Blue, injected subcutaneously, delays the production of liver cancer in rats by p-dimethylaminoazobenzene included in the diet.

A. J.

Azo Dyes. V— Dyes obtained from NN'-Bisaminophenylbenzidine. K. Marata and K. Harada. Bull. Fac. Eng., Hiroshima Univ., 3, 217-221 (1954): Chem. Abs., 49, 2737 (25 Feb. 1955).

2:2'-(4:4'- Diphenylenedimino) bis (3-nitrobenzenesulphonic acid), obtained from 1-chloro-4-nitro-2-benzenesulphonic acid and benzidine, was reduced with Fe powder
to 6:6'-(4:4'- diphenylenediimino) dimetanilic acid.
This was soluble in water and was coupled by ordinary
methods with naphthionic acid, Freund acid, amino-G
acid, J acid, J acid, Schaeffer's acid, croceic acid, R acid,
G acid, and H acid. The resulting acid dyes had better
fastness to light, water, washing, soda ash, and chlorine
than dyes made by coupling the same acids with benzidine
or tolidine. The dye from J acid had affinity for cellulose,
and that from naphthionic acid with change of pH gave
a colour change similar to that of Congo Red. C. O. C.

Red Organic Pigments. I—Rate of Decomposition of Diazo Compounds of the Benzene and Naphthalene Series. M. Mori. J. Chem. Soc. Japan, Ind. Chem. Soct., 57, 269-271 (1954); II—Physicochemical Properties of Various Kinds of Azo Dyes—1. Ibid., 271-273; III—2. Ibid., 273-275; Chem. Abs., 49, 4298 (25 March 1955).

I -- Measurements of the rate of decomposition at 1 — measurements of the table of constraints of 0.5, 0.20, 0.40, 0.50, and  $0.0^{\circ}$  c. are given of 0.08n. aq. soln. of the diazonium chlorides of aniline, p-nitroaniline, p-nitro-o-toluidine, m-nitro-p-toluidine, 2-sulpho-4-nitro-aniline, p-toluidine-m-sulphonic acid, c-amino-benzoic acid, 5-amino-2-chloro-4-sulphotoluene, 1-naphthylamine, and 2-amino-1-naphthalenesulphonic acid. Tables are given showing the decomposition constant (K) at each temperature, the temperature coefficient of K, and the activation energy for decomposition.

II—The spectro-reflective index measured by the Pulfrich photometer is given for 150 azo dyes.

III— The fastness of 150 azo dyes to light, heat, acids, alkalis, CS<sub>p</sub>, benzene, acetone, ethyl alcohol, etc. are tabulated.

C. O. C.

Azo Dyes derived from 2-Methylbenzothiazoliam Dyes derived from 2-Methysbentzonnatzonam Salts. II—Preparation of Monoazo Dyes. H. Wahl and M. T. L. Brio. Bull. Soc. chim. France, 1277-1281 (1954); III—Preparation of Diazo Dyes. Ibid., 1281-1282: Chem. Abs., 49, 3537 (10 March 1955).

I—e-Chloronitrobenzene is treated with Na<sub>2</sub>S<sub>2</sub> and then oxidised with H<sub>2</sub>O<sub>2</sub>, and the product dissolved in a mixture of acetic acid and acetic anhydride and treated with Zn, to give a 67% yield of 2-methylbenzothiazole. with 2n, to give a 6.7% yield of 2-methylpenzothnazors. This is dissolved in benzene (149 g. in 250 ml.) and dimethylsulphate added drop by drop (126 g.), to give a quantitative yield of 1.2-dimethylbenzothiazolium methosulphate. This (27-5 g.), refluxed 3 hr. with pond-capture (15 g.) and piperidine in ethyl alcohol (10 drops in 300 ml.), gives a 40% yield of 2-policy the control of the contr dimethylaminoanilinomethylene-1-methylbenzothiazolium methosulphate. This (4 g.) mixed with C<sub>6</sub>H<sub>5</sub>NH·NH<sub>3</sub> (1 g.) in 5x-HCl (100 ml.) for 4 hr. and then heated for 2 hr. yields 2-phenylazomethylene-1-methylbenzothiazoline hydrochloride. 2-p-Nitrophenylazomethylene-l-methylbenzothiazoline and 2-p-chlorophenylazomethylene-l-methylbenzothiazoline hydrochlorides can be similarly prepared.

II- The above benzthiazolines treated with diazonium salts yield compounds of formula-

2-phenylazomethylene-1-methylbenzothiazoline Thus (26 g.) in pyridine (200 ml.) when treated at  $0^{\circ}$ c. with  $C_cH_5\cdot N:N\cdot Cl$  (0·1 mol.) yielded a compound of the above formula with X and Y both = H. C. O. C.

Naphthodioxan, some Derivatives and Azo Dyes—II. P. M. Heertjes, A. M. ter Horst, and J. M. Persijn.

Rec. Trav. chim., 74, 31-40 (Jan. 1955).

5-Amino- (I), 6-amino- (II), 10-nitro-5-amino- (III), and 10-acetylamino-5-aminonaphthodioxan (IV) have prepared by conventional means from 5-nitroand 5:10-dinitro-naphthodioxan. Naphthodioxan-7-suland 5:10-dimero-naphthodioxan. Naphthodioxan-7-sui-phonic acid is obtained by condensing 2:3-di-hydroxy-naphthalene-6-sulphonic acid with ethylene dichloride, naphthalene-6-sulphonic acid with ethylene dichloride, and from this two monoaminonaphthodioxan-7-sulphonic acids (V) and (VI), of unknown orientetion, have been prepared. From these compounds the following azo dyes have been formed: I→R acid (VII), I→acetyl-H acid (VIII), I→J acid (IX), I→1-p-sulphophenyl-3-methyl-5-pyrazolone (X), II→VII, III→VII, III→X, IV→VII, IV→X, V→VII, IV→X, IVI→VII, III→X, sulphanilic acid→I, 1-aminonaphthalene-4:8-disulphonic acid→I, and metanilic acid→I aphenyl-J acid. The nonoazo dves can be apoliced. acid→I-phenyl-J acid. The monoazo dyes can be applied to wool from an acetic acid dyebath, and the disazo dye shows affinity for cotton. Fastness properties of all dyes are rather poor.

Action of Light on some Organic Colouring Matters. IV—Inhibition of Fading by Copper Sulphate. C. M. Desai and B. K. Vaidya. J. Indian Chem. Soc., 31, 717-719 (Sept. 1954).

The fading by sunlight of 0.002% soln. of Naphthol ellow, Crystal Violet, Chrysoidine, Brilliant Green, Malachite Green, Rosaniline, aminoazobenzene, and pierio acid containing copper sulphate up to  $40 \times 10^{-9} \rm M$ . has been measured colorimetrically. Increasing the concn. of copper increases the induction period. With azo dyes, fading is inhibited at low, and accelerated at high, concn. of copper sulphate.

o-Quinones. VII-Quinoneimines as Intermediates in the Reaction of Fremy's Salt with Aromatic Amines. L. Horner and K. Sturm. Chem. Ber., 88, 329–338 (March 1955).

Primary aromatic amines with occupied (e.g. p-toluidine and m-4-xylidine) or free (e.g. o-toluidine) para position are converted by Fremy's salt (potassium nitrosodisulphonate) into o- and p-quinoneimines respectively, which immediately add a further molecule of amine and are dehydrogenated to p- and o-quinone-anils respectively. o-4-Xylidine and m-toluidine gave no definite product. 5.4-Xylidine and m-totaltine gave no definite product. The amino group of mesidine was replaced by a nitrogo group in 47% yield, but m-2-xylidine gave the analogous product in only 6% yield, 25% of the p-quinoneimine and 40% of the p-quinone being formed. N-Methylaniline was transformed into p-N-methyl-N-phenylamino-oquinone, and phenthiazine into phenthiazone.

Indophenols for Sulphur Dyes. VIII—Behaviour of p-Nitrosophenol in Sulphuric Acid. O. Manabe, T. Suzawa, and H. Hiyama, J. Chem. Soc. Japan, I. Chem. Con. Japan, J. Chem. Soc. Japan, Japan, J. Chem. Soc. Japan, Japan, Japan, J. Chem. Soc. Japan, J Ind. Chem. Scotn., 57, 326-328 (1954): Chem. Abs., 49, 4298 (25 March 1955).

The absorption spectrum of p-nitrosophenol in 75% H<sub>2</sub>SO<sub>4</sub>, measured by a Beckmann spectrophotometer, showed strong absorption at 388 m $\mu$ . and slight absorption at 302 m $\mu$ . The van't Hoff dissociation index of p-nitrophenol, calculated from the values of the m.p. depression of the p-nitrosophenol-H<sub>2</sub>SO<sub>4</sub> system, was 2. It was inferred from consideration of the experimental results that the dissociation of p-nitrosophenol in H<sub>2</sub>SO<sub>4</sub> would

$$HO$$
NO +  $H_8SO_4 \rightleftharpoons O$ = $N \cdot \stackrel{\circ}{O} \cdot H + HSO_4$  $\stackrel{\circ}{H}$ C. O. C.

theses of Organic Fluorescent Compounds. XVII—Syntheses of Water-soluble Fluorescent Compounds by Sulphomethylation of Monoamino Compounds and their Fluorescent Brightening Effect. R. Oda, Z. Yoshida, and Y. Shimada. J. Chem. Soc. Japan, Ind. Chem. Sectn., 55, 782-784 (1952); XVIII—Syntheses of Water-soluble Com-pounds by Sulphomethylation of Diamino Compounds and their Fluorescent Brightening Effect. Ibid., 784-786; XIX—Syntheses of Fluorescent Compounds by Cyanomethylation and their Fluorescent Brightening Effect. 1bid., 786-XX—Syntheses of some Fluorescent Resins. Z. Yoshida, S. Inoue, and R. Oda. *Ibid.*, 56, 44–45 (1953): Chem. Abs., 49, 3915 (25 March 1955).

XVII—Formation of R·NH·CH<sub>2</sub>·SO<sub>2</sub>Na from R·NH<sub>3</sub>, HCHO, and NaHSO<sub>2</sub> is termed sulphomethylation. Measurement of the fluorescent intensities of 1- and 2-HoN.C10H4.NH.CH2.SO3H, Na 1-carbazolylaminomethanesulphonate, 6-methyl-2-p-sulphomethylaminophenylbenzothiazole, etc. showed that the solid compounds had medium to strong violet or bluish-violet fluorescence. 6-Methyl-2-p-sulphomethylaminophenylbenzothiazole is a fluorescent brightening agent for wool. **XVIII**—Comparison of the fluorescence of (2-R- $C_4H_4CH:$ )<sub>1</sub> (1), (4- $RC_4H_4CH:$ )<sub>2</sub> (II), (4- $RC_2H_4$ )<sub>2</sub> (III), (4- $RC_2H_4$ )<sub>2</sub> (III), (4- $RC_3C_4H_4$ )<sub>3</sub>, (4- $RC_3C_4H_4$ )<sub>3</sub>, (1- $RC_3C_4H_4$ )<sub>4</sub>, (1- $RC_3C_4H_4$ ), (1-

XIX—1·C<sub>16</sub>H<sub>1</sub>N(CH<sub>2</sub>CN)CH<sub>2</sub>SO<sub>3</sub>H, 6·CH<sub>2</sub>·2·{4·{N-cyanomethyl)sulphomethylamine}} phenyl)benzothiazole, and 2·(cyanomethylamino)-2·(sulphomethylamino)-stilbene when solid have strong violet to blue fluorescence. They have good solubility, fastness to light and, with the exception of the first named, a good fluorescent brightening effect on wool.

XX— The resins obtained by polymerising 2-(2-hydroxyethyl)benzimidazole, 2-(2-hydroxyethyl)naphth-[1,2]-imidazole and 2:1-CH<sub>3</sub>OC<sub>18</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>OH and copolymerising p-C<sub>8</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> and 3:1:4-CH<sub>2</sub>C<sub>4</sub>H<sub>3</sub>(COOH)<sub>2</sub> fluorescender the mercury lamp but not in sunlight, the intensity of the fluorescence being weak to medium. C. O. C.

Dyes for Synthetic Fibres. I—Blue Sulphur Dyes containing a 2-Hydroxyethyl Group. N. Kroki, A. Katayama, and K. Konishi. J. Chem. Soc. Japan., Ind. Chem. Sectn., 57, 291-293 (1951): Chem. Abs., 49, 4298 (25 March 1955).

Derivatives of (2-hydroxethyl)allylamine-indophenol were obtained by treating the corresponding allylamine with p-nitrosophenol in conc. H<sub>2</sub>SO<sub>4</sub> at 0-5°c. for 3 hr. (Hydroxyethyl)methyl derivatives of indophenol were also obtained by oxidation condensation of p-amino-NN-diallylamiline with 1-naphthol in presence of K<sub>2</sub>Cr<sub>2</sub>Or, and acetic acid. The fastness to washing, light, and rubbing of these blue dyes was ascertained. C. O. C.

Acid Anthraquinone Dyes. III— Acid Dyes derived from 1-Amino-2:4-dibromo-8-anthraquinone-sulphonic Acid. K. Murata and K. Harada. Bull. Fac. Eng., Hiroshima Univ., 3, 201-203 (1954); IV— Vat Dyes obtained from Benzanthraquinone Ibid., 205-209: Chem. Abs., 49, 2738 (25 Feb. 1955). III— Syntheses of 10 dyes are described, and data given about their dyesing and featness representations.

given about their dyeing and fastness properties.

IV—Syntheses from 4-bensylaminobenzanthraquinone by condensation with halogeno derivatives are given of 24 vat dyes together with data on yields, hue, etc.

C. O. C.

Vat Dyes of the Pyrazoloanthrone Series. V—Selfcondensation of 5-Chloropyrazoloanthrone. T.
Maki and H. Seki. Bull. Chem. Soc. Japan, 27,
613-617 (Dec. 1954).

5-Chloropyrazoloanthrone (I) is prepared from 1-amino-5-chloroanthraquinone by diazotisation, treatment of the neutral diazo compound with sodium sulphite to give the diazosulphonate, reduction of this to potassium 5-chloroanthraquinony1-1-hydrazinesulphonate by the action of potassium bisulphite, and ring closure to I. This method gives a high yield of m.p. 305-306°c. (cf. m.p. 298°c., Bradley and Geddes, J.C.S., 1633 (1952)). On heating I with sodium carbonate and copper in boiling nitrobenzene for 24 hr. a product is obtained which, from its chlorine content, is supposed to be a mixture of triand a tetra-molecular condensates produced by elimination of HCl from the imino nitrogen of I and the 5-chlorine atom of further molecules of I. In the presence of potassium carbonate almost complete conversion to the tetramolecular condensation product is claimed. Similar condensation occurs between I and 3-bromobenzanthrone and between I and 1-aminoanthraquinone.

A. J.

Vat Dyes of the Acenaphthene Scries. VI—Derivatives of Acenaphthene Violet. T. Maki and H. Hashimoto. Bull. Chem. Soc. Japan, 27, 602-605 (Dao. 1954).

The following derivatives of Acenaphthene Violet (I: X = H) (cf. 4bid, 25, 411 (1952)) have been prepared—
(i) pp'-dichloro (I: X = Cl) by condensation of 4-chloro-

1:2-phenylenediamine with 3:4:9:10-perylenetetracarboxylic anhydride (II), a brilliant violet dye; (ii) dibenzo (III) by condensation of II with 1:2-naphthylenediamine, a violet-blue dye; (iii) pp'-dimethoxy (I: X = 0-CH<sub>3</sub> by condensing II with 4-methoxy-2-nitroaniline to give the bis-p-methoxy-o-nitrophenyl-diimide of II, followed by reduction of this to the corresponding diamino compound and ring closure. All these dyes have good fastness properties, particularly to light, when dyed on cotton. Max. light fastness (8) is shown by unsubst. I. Reflectance curves of 1% dyeings on cotton are given and analysed. The dichloro deriv. has the highest colour purity.

Synthesis of 4- and 6-Bromoindole and of 4- and 6-Aminoindole. H. Plieninger, T. Suehiro, K. Suhr, and M. Decker. Chem. Ber., 88, 370-376 (March 1955).

Structure and Properties of Certain Polycyclic Indolo and Quinoline Derivatives. VIII—Derivatives of 1:6-Dioxojulolidine. J. T. Braunholtz and F. G. Mann. J.C.S., 393-398 (Feb. 1955). The bisphenyl- and bisdiphenyl-hydrazones of 1:6-dioxojulolidine (I) undergo the Fischer indolisation to the corresponding di-indolojulines, the former being table of the corresponding di-indolojulines, the corresponding di-indolojulines, the corresponding di-indolojulines, the corresponding di-indolojulines, the corresponding di-indolojulines di-indolojulines di-indolojulines di-indolojulines di-indolojulines di-indolojulines di-in

The bisphenyl- and bisdiphenyl-hydrazones of 1:6-dioxojulolidine (I) undergo the Fischer indolisation to the corresponding di-indolojulines, the former being stable only in its orange salts, e.g. the hydriodide, thioxyanate, and pierate, where salt formation occurs on the central N atom, and the latter being isolable but very unstable in contact with organic solvents. y-Indole formation is not observed. I when condensed by the Pfitzinger reaction with isatin in alkaline soln. gives the purple diquinolinojulinedicarboxylic acid (II), which on heating followed by decarboxylation gives the orange diquinolinojuline (III), converted by hot HCl into an isomeric red base.

Cyanine Dyes derived from 1:6-Dioxojulolidine. J. T. Braunholtz and F. G. Mann. J.C.S., 308-401 (Feb. 1955).

7-Methyl-1:6-dioxojulolidine (I) condenses with 2-methylbenzothiazole ethiodide to give the oyanine iodide (II), but the corresponding deriv. could not be obtained from 1:6-dioxojuloline. The latter, however, readily condenses with p-dimethylaminobenzaldehyde and p-nitroso-dimethylamiline to give mero-cyanines of type (III). Other deriv. are briefly described.

H. H. H Synthesis of Cyanine Dyes by Condensation of p-Diethylaminobenzaldehyde with appropriate Heterocyclic Compounds. M. Q. Doja and A. K. Sinha. J. Indian Chem. Soc., 31, 735-740 (Oct. 1954).

6-Amino, 6-chloro, 6-bromo, and 6-iodo-2-p-diethyl-aminostyrylquinoline ethiodide are obtained by condensing the ethiodides of the appropriate bases with p-diethyl-aminobenzaldehyde in absolute ethanol in the presence of piperidine. The dyes have poor light and washing fastness and are not satisfactory as photographic sensi-

Trinuclear Cyanine Dyes. V—Decomposition of Oxacyanines. Y. Rishiki. J. Soi. Research Inst. Oxacyanines. Y. Hishiki. J. Soi. Research Inst. (Tokyo), 48, 130-142: Chem. Abs., 49, 4295 (25 March 1955).

N-CH<sub>2</sub> and N-C<sub>2</sub>H<sub>5</sub> derivatives of trinuclear oxacyanine diodide dyes are changed to their iodide hydroxide dyes by weakly basic substances in alcoholic solution, to their dihydroxide dyes in aquous NH<sub>4</sub>OH, and finally to colourless substances which are monohydroxide compounds. N-CH<sub>3</sub> and N-C<sub>2</sub>H<sub>5</sub> derivatives of oxa-carbocyanine iodides are converted into their hydroxide dyes with alkali. The mechanisms involved are discussed and a large number of examples given. C. O. C.

Pyrroletrimethin Dyes—II. A. Treibs and E. Herrmann. Annalen, 592, 1-10 (March 1955).

Experiments on the mechanism of formation of such dyes are recorded and discussed.

III—A. Treibs and K. Hintermeier. Ibid., 11-25.

2-Acetylpyrroles carrying a substituent in position 5 condense with themselves under the influence of carboxylic acid chlorides to give dyes of the probable constitution

2:4-Dimethyl-3-ethoxycarbonylpyrrole reacts with acetyl chloride ultimately to yield a tripyrryltrimethin dye, and the mechanism of this and analogous reactions is meso-Substituted dipyrrylmethins are also discussed. prepared.

Paramagnetic Resonance in Copper Phthalocyanine.
J. E. Bonnett and D. J. E. Ingram. Nature, 175, 130-131 (15 Jan. 1955).

Signals from dil. crystals of Cu phthalocyanine have been observed at 1.25 cm. and 8-mm. wavelengths, and at temp. down to 20°x. The hyperfine splittings and g values have been measured. Since X-ray analyses have afforded an acourate picture of the crystal structure of metal phthalocyanines, it has been possible to correlate the paramagnetic resonance measurements with the actual directions of the copper-nitrogen bonds. The findings are discussed in some detail.

J. W. D.

Mechanism of the Photoreduction of certain Dyes in presence of Chlorophyll and Related Substances as Sensitisers. V. B. Evstigneev and V. A. Gavrilova. Doklad? Akad. Nauk S.S.S.R., 98, 1017-1020 (21 Oct. 1954).

In the photoreduction of Safranine T, riboflavin, or Methyl Red by ascorbic acid in presence of phaeophytin a or b or magnesium phthalocyanine as sensitiser at low , features in the absorption spectrum associated the sensitiser remain unchanged until the dye is

completely converted into its leuco form, when changes associated with the formation of the primary, labile reduction product of the sensitiser begin to occur. It is inferred that the reversible formation of this reduction product is the basis of the consitising effect. vidence that the mechanism of the sensitising action of chlorophyll is similar. A. E. S.

Inhibition of Photosynthesis by Oxygen. I. Ya. Shlyapintokh. Doklad? Akad. Nauk S.S.S.R., 99, 141-144 (1 Nov. 1954). It is considered that the inhibiting action of oxygen on photosynthesis in plants, often attributed to its effect

on enzymic dark reactions, is in fact due to its effect on the photosensitising action of chlorophyll. The theoretical basis of such effects is discussed, and a model experiment is carried out in which the retarding effect of molecular oxygen on the eosin-sensitised autoxidation of butyraldehyde is detected and measured. A E S

Infrared Spectra and cis-irans Configurations of Some Carotenoid Pigments. K. Lundo and L. Zechmeister. J. Amer. Chem. Soc., 77, 1647-1658 (20 March 1955).

Consideration of the infrared spectra in relationship to cis-trans configurations of some representatives of the following stereoisomeric sets- β-carotene, zeaxanthin, lycopene, a-carotene, y-carotene, dimethylcrocetin, and methylbixin—has resulted in confirmation of some configurations proposed earlier on the basis of spectral phenomena, observed in the visible and ultraviolet regions, while some others have had to be revised. C. O. C.

Ultraviolet Absorption Investigation of Melanins. W. D. Stein, Nature, 175, 472 (12 March 1955).

The melanin of the ox-choroid is identical with that

of black human hair, while red human hair contains a pigment related to, but not identical with, these melanins.

Yellow Hydrous Ferric Oxide. S. R. Gupta and S. Ghosh. Proc. Natl. Acad. Sci. India, 21A, 3-10 (1952) 'Symposium on Chem. Hydrous Oxides".

PATENTS

Cyan Colour Formers. ICI. BP 727,693 Compounds of formula

= Alk of > 4C;  $R^2$  = Alk of < 5C), e.g. 1-hydroxy-4sulpho-2-naphthoyl-N-methyl-N-octadecylamide, used as cyan colour formers in colour photography yield bluer images than do 1-hydroxy-2-naphthoylamides but have the same valuable water-soluble, non-diffusing properties.

Arylides of o-Carboxyhydroxy-1:2:3-benzotriazoles-Azoic Coupling Components. American Cyanamid Co. USP 2,675,376 Arylides of e-earboxyhydroxy-1:2:3-benzotriazoles

(OH and [CONH-Aryl are ortho to one another) are coupling components for azoic dyes and pigments, particu-Thus 5-hydroxy-1:2:3-benzotriazole treated with  $K_3CO_3$  and  $CO_3$  under pressure at 180–190°c. for 16 hr. to give the 4-carboxy compound. Treatment with aniline and  $PCI_3$  in chlorobenzene converts this into the anilide, which couples with diazotised 2:5-dichloro-aniline to give a brownish-orange. E.S.

Metal(Chromium)-complex Monoago Dyes for Wool, etc. Gy.

BP 727,585
The complexes containing at least 1 atom of chromium to 2 mol. of monoago compounds-

(R = H, Hal or CH<sub>5</sub>) dye wool, silk and nylon from neutral or weakly acid baths. The coupling component is prepared from \$O\$-acyl-l-naphthol-3:6-disulphonic acid by treatment with PCl<sub>5</sub> to give the disulphonic acid chloride, followed by treatment with methylamine, and alkaline hydrolysis of the \$O\$-acyl group. It is coupled for instance with diazotised 2-amino-4-chloro-5-nitrophenol, and the mono-azo compound so formed heated at \$O\$-100°C. with ammoniacal ammonium disalicylate chromate to give the chromium complex. The dye so formed is mixed with an anionic dispersing agent and a little trisedium phosphate, and gives greenish-blue on wool.

E. S.

Yellow Monoazo Acid Dyes of Good Fastness to Chlorine. Gy. Monoazo dyes—

(Alk of 8–12C; Aryl is a benzene or naphthalene series radical; one SO<sub>3</sub>H group is also present in the molecule) dye wool from a neutral or weakly acid bath yellows of good fastness to chlorination. Thus the monoazo dye anthranilis acid s-decyl ester->1-(2'-chloro-5'-sulphophenyl)-3-methyl-5-pyrazolone gives a greenish yellow.

Monoazo Dyes for Wool and Nylon. Gy. BP 725,814 1-Amino-8-naphthol-3:6(or 4:6)-disulphonic acids (or their O-acyl derivatives) are diazotised and coupled with dibenzylanilines—

 $(Y=H\ or\ CH_3;\ Z=H,\ Alk,\ or\ OAlk)$  followed if necessary by de-acylation, to give monoaxo dyes applicable to wool or nylon from neutral or weakly acid baths. Thus O-tosyl-lamino-8-naphthol-4:8-disulphonic acid is diazotised and coupled under weakly acid conditions with an alcoholic solution of NN-dibensylaniline. The tosyl group is removed by stirring with a NaOH at 80-85°c. The product dyes wool bluish red from a weakly acid bath. E. S.

Chromable Monoazo Dyes. Ciba. BP 714,166
Diazotised derivatives of o-aminophenol are coupled with 1-sulphoacetylamino-7-naphthol, i.e.—

to give chromable wool dyes, particularly suitable for the after-chroming process. Thus 1-amino-7-naphthol is converted into its N-chloroacetyl compound by treatment at below 30°c. with chloroacetyl chloride in aqueous acetone containing sodium acetate. Boiling with aq. sodium sulphite in presence of acetone then gives 1-sulphoacetyl-amino-7-naphthol, which is alkali-coupled with diazotised 2-amino-5-nitrophenol. The monoace dye so formed dyes wool red-violet, converted to blue-green by after-chroming.

Orange to Claret Azoic Dyes for Acetate and Nylon.

FH. BP 727,443

Oranges to clarets fast to cross-dyeing are produced on acetate and polyamide fibres by applying a diazotisable

(Hal, Alk, OAlk, or Oaryl may be present in the benzene nucleus;  $\mathbf{R}^1$  and  $\mathbf{R}^2 = \text{aralkyl}$ , aryl or cycloalkyl radicals, or  $\mathbf{NR}^1\mathbf{R}^3 = \mathbf{a}$  piperidine radical) together with an arylide of 3:2-hydroxynaphthoic acid containing in the arylide residue OAlk alone or with Hal, Alk or other OAlk groups, followed by diazotisation on the fibre and development in a hot bath. Thus 2:5-dimethoxyaniline-4-sulphonpiperidide with 3:2-hydroxynaphtho-4'-methoxy-2'-methylanilide gives clarets on acetate or polyamide fibres. E. S.

Metal(Chromium)-complex Blue Azo Dyes for Wool and Nylon. DuP. USP 2,671,081 Monoazo compounds—

(Alk = CH<sub>2</sub> or C<sub>2</sub>H<sub>5</sub>; Y and Z = H, SO<sub>2</sub>NH<sub>2</sub>, SO<sub>2</sub> NHCH<sub>3</sub>, or SO<sub>2</sub> NHC<sub>2</sub>H<sub>5</sub>; Z is in a  $\beta$ -position in the ring indicated) are converted into chromium complexes containing 1 atom of Cr to 2 mol. of monoazo compound. During the chroming, preferably carried out in presence of ethylene glycol, formamide, or acetamide, the OAlk group orthoto the azo link is dealkylated. The products dye wool and nylon blue from a neutral bath. Thus the azo compound 2.5-dimethoxyaniline  $\rightarrow \beta$ -naphthol is stirred at 140–160°c, with chromium acetate in ethylene glycol and sufficient NaOH to maintain an alkaline reaction throughout. E. S.

Metal(Chromium and Cobalt)-complex Dyes for Wool and Nylon. Ciba. BP 726,342

The chromium and cobalt complexes of the monoazo compound 2-aminophenol-5-sulphonamide→β-naphthol containing l atom of metal to 2 mol. of monoazo compound are soluble in water and dye wool and nylon from neutral or weakly acid baths. Metallisation is carried out under weakly acid to alkaline conditions, in presence of hydroxy-carboxylic acids, e.g. lactic or salicylic acid when chromium is used, whereas the cobalt complex may be formed simply by using cobalt chloride, sulphate or hydroxide. The cobalt and chromium complexes dye wool bluish bordeaux and bluish violet respectively. E. S.

Chromable Monoazo Blues for Wool. Gy.

BP 726,346

Diazotised 2-amino-6-nitrophenols, containing in the 4-position an alkyl or cycloalkyl substituent of 4-8 C, are coupled with 1-naphthol-3:6-disulphonamide to give blue dyes which may be chromed in substance, on the fibre or in the dyebath. Both the ummetallised and metallised dyes exhaust well on to wool, nylon, etc., from a neutral or weakly acid bath. The diazo components are made by dinitration of suitable p-alkyl- or p-cycloalkyl-phenols, followed by reduction of one of the nitro groups with e.g. Na<sub>8</sub>S. Thus p-tert.amylphenol gives 2-amino-6-nitro-4-tert.amylphenol, which is diazotised and coupled with 1-naphthol-3:6-disulphonamide. The product dyes wool violet, converted to blue by afterchroming; or it may be applied by the metachrome process, or converted in substance into its chromium complex by heating at 90-100°c. with aq. ammonium disalicylato chromate. E. S.

Yellow Mono- and Dis-azo Dyes from 6-Amino-2-cyanamino-4-hydroxypyrimidine. FBy. BP 727,045

Azo dyes made by coupling 6-amino-2-cyanamino-4hydroxypyrimidine—

with diazo and tetrazo compounds of the benzene series containing at least one  $SO_3H$  or COOH group dye wool, cellulose fibres, and paper yellow. Thus tetrazotized

3:3'-diaminodiphenylurea4:4'-disulphonie acid couples under alkaline conditions in position 5 to give a greenish yellow for cotton and regenerated cellulose fibres. E. S.

Dis- and Poly-azo Direct Cotton Dyes. S. BP 726,726 Two mol, of an aminoazo compound—

(R = benzene or naphthalene series radical which may have a group capable of giving a metal-complex ortho to the N:N link), or 1 mol. of such a compound and 1 mol. to the N:N link), or I mol. of such a compound and I mol. of any other aminoaso compound, are condensed with I mol. of a dicarboxylio acid dihalide Hal-CO-CX:CH-COHal (Hal = Cl or Br; X = H, Cl, Br, or CH<sub>2</sub>), to give direct cotton dyes, some of which may be metallised in substance or on the fibre. Alternative methods of building up the dye molecule required are available. When two different aminoaso compounds are used the molecular proportions employed may sometimes with advantage be other than 1:1. Thus a mixture of the aminoances of compounds. aminomonoazo compound-

(30.0 parts) and the aminodisazo compound-

(26-8 parts) is treated with fumaryl dichloride in presence of NaOH temperature. The products dye and No<sub>2</sub>CO<sub>3</sub> at room temperature. The products dye cellulose fibres yellow-orange, which may be aftertreated with copper compounds. E. 8.

Fluorescent Brightening Agents. General Aniline USP 2.674.604

Condensing 1 mol. of 4:4'-diamino-2:2'-stilbene disulphonic acid with an excess of about 2 mol. of a dihydrobenzofurancarboxylic acid halide yields fluorescent brightening agents of formula-

 $(R^1 = Alk, preferably CH_9; R^8 = H or Alk, preferably CH_9; M = H or cation).$  C. O. C.

Azodiphenyl-Anthraquinone Vat Dyes. DuP. BP 726,797

USP 2,601,100 (J.s.D.C., 69, 169 (1953)) is extended to cover compounds containing additional groups, e.g. > 2 SO<sub>2</sub>M (M = alkali metal), which are also yellow vat dyes. Thus the dye is prepared by first heating 4:4'-azodiphenyl-4":4"-dicarboxylic acid with SOCl<sub>3</sub> in nitrobenzene containing pyridine at 98-100°c, for 12-20 hr. After removing excess SOCl<sub>3</sub> by aerating, and cooling to 40°c, 1-amino-2-chloroanthraquinone is added and the whole heated at 140°c, for 6 hr. 2-Aminoanthraquinone-3-sulphonic acid (Na salt) is next added at 25°c, and after heating at 208°c, for 1 hr., Na<sub>3</sub>CO<sub>3</sub>, potassium acetate and copper acetate introduced. The whole is finally heated at 208-210°c, for 2·5 hr. R. K. F.

Brominated "Dianthrone Ethylenes". CFM. BP 726,429

The red to violet "dianthrone ethylenes

of BP 299,972 (J.S.D.C., 45, 89 (1929)) are treated with of Br 299,912 (J.S.D.C., 45, 59 (1929)) are treated with bromine in chlorosulphonic acid containing a small amount of iodine, to give blue compounds which are no longer vat dyes. They are intermediates which are converted into accdianthrone vat dyes, e.g. by treating with H<sub>2</sub>SO<sub>4</sub> or heating in nitrobenzene. Thus the violet dye obtained from anthrone and glyoxal is dissolved in chlorosulphonic acid containing iodine, and bromine added at 30°c. The product is crystallised from o-dichlorobenzene. When heated at 200-208°c, in nitrobenzene a brown vat dye is produced.

10-Benzoyl-3:7-bis (dimethylamino) phenothiazine.

National Cash Register Co.

BP 725,275

Methylene blue is reduced with e.g. sodium hydrosulphite and aq. NaOH and benzoyl chloride added slowly. After filtering, the product is dissolved in a non-plan solvent of the control of the polar solvent, e.g. toluene, containing fullers earth. The solution after filtering is evaporated to obtain the 10-benzovl derivative-

R. K. F.

Dry Sulphuric Acid Esters of Leuco Vat Dyes. Ciba

Sulphuric acid esters of leuco vat dyes are readily obtained as dry stable powders by spray drying their solutions. COC

Co-precipitated Complex of Silica, Titanium Dioxide and Calcium Sulphate— White Pigment. Northern Minerals.

USP 2.674.641

The complex consisting of CaSO<sub>4</sub> (136 parts by wt.), SiO<sub>2</sub> (60) and TiO<sub>3</sub> (65-600) obtained by the co-precipitation of its components has much better hiding power than TiO<sub>3</sub> or TiBa or TiCa pigments. Any method of producing the co-precipitate may be used. C. O. C.

Resin-coated Clay Pigment. Edgar Brothers USP 2,674,587

A pigment suitable for filling or coating paper or for use with rubber is produced by spray drying a mixture of clay and an aqueous dispersion or solution of a urea-melamine- or phenol-formaldehyde condensate. C. O. C.

Organic Dyes in Photography (IX p. 363).
Bismuth Oxycarbonate or Oxysalicylate for producing
Pearlescent Effects in Plastics (XIII p. 367).

#### V-PAINTS; ENAMELS; INKS

Agents for Dispersing Pigments in Mineral Oil.

American Cyanamid Co.

Mineral Oil. American Cyanamid Co. USP 2,675,320 Compounds of formula R'OOC·CH<sub>2</sub>·CH(COOR<sup>3</sup>)SO<sub>2</sub>M (M = a strongly basic metal; R<sup>1</sup> and R<sup>2</sup> = Alk or residue

of incompletely esterified glycerine and together contain > 17C) e.g.

CH, CH CH,

CH, C.H.

CH<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>·CH·CH<sub>3</sub>·CH<sub>3</sub> · CH·OOC·CH<sub>2</sub>

CHa(CHa)a CH-CHa-CHa · CH-OOC-CHSOaNa

CH<sub>2</sub> C.H.

CH, CH-CH,

are excellent agents for making dispersions of organic or inorganic pigments in mineral oil. USP 2,675,321

Compounds of formula-

(R1, R2 and R3 = Alk and together contain > 17 C; alkali forming metal; n = valency of M) e.g. dihexyl dithiophosphato-3:5:5-trimethylhexyl barium succinate, have similar properties.

Emulsion Paints of Improved Freeze-Resistance. Firestone Tire & Rubber Co. BP 726,463 Increase in viscosity of emulsion paints on repeated freezing is minimised by adding hydrazine hydrate or a substituted hydrazine (approx. 2% on the dry polymer) to the polymer latex or the finished paint.

Increase in viscosity of emulsion paints on repeated freezing is minimised by adding a polyalkylene polyamine, e.g. diethylene triamine (approx. 1% on the dry polymer).

Polybutadiene Emulsion Paints. Standard Oil Development Co. BP 726.331

Hard flexible colour-stable coatings of improved scrubresistance are deposited from an emulsion paint (45-55% total solids) containing a polybutadiene latex (35-40%), a pigment (17-20%), kaolin (1-3%), soyabean protein (1-3%), a metallic drier (0-01-0-03%), and methyl cellulose (0.18-0.22%). The latter suitably raises the viscosity without causing instability or gelling. E.C.

#### VI-FIBRES; YARNS; FABRICS

Static Electricity in Textiles. K. H. Barnard. Amer.

Dyestuff Rep., 44, P111-P113 (14 Feb. 1955).
Problems caused by static electricity, its elimination, and the evaluation of antistatic finishes are briefly dis-J. W. B.

Materials. R. Schaffer, W. D. Appel, and F. H. Forziati. Bur. Stand. J. Res., 54, 103-106 (Feb. 1955).

"brown-line" reaction on cellulosic materials at the boundary between areas wet with water and dry areas is found to occur at wet-dry interfaces involving other fibres and liquids. Examples are given and the nature of the reactions is discussed.

Cotton Quality and Fibre Properties. II— Cavitomic Cotton. H. Wakeham, H. Stickley, and N. Spicer. Text. Research J., 24, 1037-1047 (Dec. 1954).

Certain raw cottons, subjected to action of fungi, suffer deterioration on storage and are termed cavitomic Studies of fabric from such cotton show more processing waste, less end slivers, more end breakage, weaker yarns, and shorter fibre length. It is suggested that this is due to localised weakening by either microbial or enzyme action during storage.

Action of Acids on Cotton and Rayon. Melliand Textilber., 36, 261-265 (March 1955). Conditions for minimum damage from dilute acids to

cotton and viscose rayon staple can be deduced from the

data given. The effect of acids on cellulose, the kinetics of the reactions, and comparisons between D.P., breaking strength, bending load, and extension are discussed and the strength, bending load, and extension at the degree of permanganate number is compared with the degree of S. R. C.

Skin and Core of Viscose Yarn. J. F. Briggs. J. Textile Inst., 46, 7230-7231 (March 1955).

An interesting letter from a pioneer of regenerated cellulose rayons includes details of how to prepare bulk samples of skin and core.

Small-angle X-Ray Scattering Power of Cellulose Fibres. F. Stein. Trans. Faraday Soc., 51, 430-441 (March 1955).

The variation of small-angle X-ray scattering power of ramie and jute fibres with moisture content has been studied. Results are explained by assuming that water is absorbed in amorphous regions. Swelling of amorphous regions reaches a limit at humidities near saturation, and any additional water held by the fibres in the wet state must be in the form of droplets of linear dimensions  $> \sim 100$  A., located in pores or cracks in the cell or on the surface of the fibres. W. R. W. R. M.

Structure of Jute Hemicellulose. P. C. Das Gupta and B. 8 Text. Research J., 24, 1071-1072 rkar. (Dec. 1954).

3-Methylglucuronic acid is shown to be the major constituent of jute hemicellulose, by methylation of methylaldobiurcnic acid from jute hemicellulose I, reduction by lithium aluminium hydride, hydrolysis of the product, and detection of 2:3:4-trimethylglucose by paper partition chromatography. W. R. M.

Studies in Fellmongering. I—Sweating of Sheep-skins. G. H. Green. J. Soc. Leather Trades Chem., 39, 20-31 (Jan. 1955).

The Lennox depilation load method has shown that a much looser wool is required for knee pulling than for pulling over a beam, this accounting for the greater difficulty in sweating the skins. A bacterium responsible for wool loosening has been isolated and characterised as a hitherto undescribed species of the genus Pseudomonas. Its inoculation on to sheepskins markedly reduced the time required for complete wool loosening by reducing the initial lag period. The bacteria which digest the wool the initial lag period. The bacteria which digest the wool root during sweating penetrate to the wool root from the flesh surface, and not down the wool folliole. During sweating, large amounts of CO<sub>2</sub> and smaller amounts of volatile acids and NH<sub>3</sub> are produced by bacterial action on the skin proteins. In the water in the skin tissues, these products are in mutual equilibrium and stabilise the skin pH at ca. 8·3. This pH is somewhat above the optimum for the growth of the unhairing bacteria, but it is not ressible to disturb the equilibrium under parmal optimum for the grown of the unnaring bacters, out it is not possible to disturb the equilibrium under normal conditions and so lower the pH. Ammonia introduced prior to or during sweating retards wool loseening unless added in large quantities towards the end of the sweating, when it completes the process by chemical means. Nematodes on the flesh surface of sweating skins are beneficial; they probably help by distributing bacteria over the skin and, later on, by keeping down the bacteria population. The source of infestation has been traced to the soak pits and supports in the sweating chambers. J. W. D.

Influence of Liquid Films on Fibre Friction. G. King. Nature, 175, 383 (26 Feb. 1955).

The adhesive force due to a film of liquid held between a cylinder and a plane increases, with reduction in the amount of the liquid, to a max. value which depends upon the tensile strength of the liquid and upon surface roughness. Results obtained using nylon filament and a polished glass cylinder, with a film of oil, illustrate the effect of oil-film thickness; with a wool fibre the roughness due to the surface scales eliminates adhesion effects. to a large extent.

Rôle of Thiol Groups in the Long-range Elasticity of Wool. R. W. Burley. Nature, 175, 510-511 (19 March 1955).

There is no increase in the number of thiol groups present when Merino wool is stretched to about 60 elongation in water at room temp. Though this suggests that hydrolysis plays little part in the stretching, the is an alternative explanation. Interchanges between thiol groups (of which a number exist in the free form in wool) and disulphide bonds may occur under the conditions of stretching, with the formation of new disulphide links and new thiol groups; the new links, since they are formed during stretching, will not oppose subsequent stretching, and they will be formed at the expense of the bonds that originally opposed stretching. Wool fibres in acid soln., or in which the thiol groups have been blocked by reaction with N-ethylmaleimide, are difficult to extend beyond ca. 37% (up to which value there is little difference from normal wool in water); the blocking reagent cannot form new cross-links, and must therefore operate by the inhibition of thiol-disulphide interchanges. J. W. D.

Structure of Silk Fibroin, N. S. Andreeva and V. I. Iveronova, Dokladý Akad. Nauk S.S.S.R., 99, 991-993 (21 Dec. 1954).

By use of a cylindrical X-ray diffraction camera and suitable fibre orientations, some new diagonal, equatorial, and meridional arcs have been found in the pattern produced by a fibre of Bombyx mori silk fibroin. Arcs identified with the second-, fourth-, and sixth-order meridional reflections are strong, weak, and very strong respectively (the first, third, and fifth orders being absent), in accord with calculations for a model based on fully extended polypeptide chains, but not in accord with the model favoured by Pauling and Corey (Proc. Nat. Acad. Soi., 39, 253 (1953)).

Yellowing of Silk. I— Causes. J. Pinte, Y. Teyssier, and P. Rochas. Bull. Inst. Text. France, (51), 7-16 (Feb. 1955).

(Feb. 1955).

Samples of silk fibroin were kept at 100°c. for 180 hr. or longer under the following conditions—(a) in a high vacuum, (b) in dry N<sub>b</sub>, (c) in water vapour diluted with N<sub>b</sub>, (d) in dry O<sub>b</sub>, and (e) in O<sub>b</sub> and water vapour. Photocolorimetric analysis and fluidity tests showed that inappreciable yellowing and chemical degradation took place in (a) and (b). Distinct yellowing and degradation took place in (a) and (b). Distinct yellowing and degradation were observed in (c) and (d), and the changes were still more marked in (e). Tests on samples containing various amounts of residual sericin showed that the presence of more than 5% increases the tendency of silk to yellow on ageing. Of samples of silk given (i) acid treatment, (ii) alkaline treatment, and (iii) oxidising treatment, only those subjected to (i) showed an increased tendency to yellow on ageing.

J. C. F.

Moisture Regain of 6,6-Nylon Continuous-filament Yarns, M. V. Forward and S. T. Smith. J. Textile Inst., 46, x158-x160 (Feb. 1955).

Moisture regains of seoured undrawn 6,6-nylon yarns are determined at 25°c. over the range 25-90% R.H. Results are in good agreement with previous findings. The presence of spinning finish has an appreciable but inconsistent effect on moisture adsorption, and results in poor reproducibility of values obtained on unscoured yarn; the best method of removing finish is by using polar liquids such as aqueous Dispersol VI or ethanol. Polar liquids swell the yarn and slightly increase moisture affinity, but strong swelling agents such as aqueous phenol cause a decrease. Filament denier has little effect on the equilibrium moisture uptake but influences the rate of uptake, which is probably associated with decrease in specific surface area. It is also found that moisture uptake overshoots the equilibrium level during sorption, the effect increasing with filament denier and being shown by both drawn and undrawn yarns; it may be associated with stresses set up in the yarn by the swelling action of the imbibed moisture. A possibly related phenomenon is the increase in equilibrium uptake of moisture by the stresses induced by high twist in drawn yarn—an example is quoted where \(\frac{1}{2}\text{-t,p.in.}\), yarn has a regain of 3.59% compared with 3.70% for 63 t.p.in.

J. W. B.

#### PATENTE

Modified Viscose Filaments. Courtsulds. BP 723,435 Viscose filaments having practically no core are produced by spinning viscose into sulphuric acid and zine sulphate in presence of 0-25-5 millimoles per 100 g. viscose of a polyalkylene polyamine having > 2 primary or secondary amino groups not directly united to one another.

W. G. C.

Artificial Filaments of Cellulose or Cellulose Derivatives of High Wet Strength. American Patents Corpn. BP 727,317

The collulose or cellulose derivative is impregnated before or during dissolution to form the spinning solution, with or without subsequently to formation of the solution, with an unsaturated fatty acid containing > 2 carbon-to-carbon double bonds, e.g. whale oil. This imparts much greater wet strength to the filaments formed from such solutions than would be obtained without such impregnation.

C. O. C.

Regenerated Protein Fibres. American Patents Corpn. BP 723,214

Threads of improved tensile properties are obtained by treating the protein prior to extrusion with a water-soluble tin salt, e.g. stannous chloride.

The protein is pre-treated with a fatty tanning substance, e.g. a highly unsaturated triglyceride fatty oil, which improves the spinning properties and makes the hardened fibre water-repellent.

W. G. C.

Improving the Wet Strength of Regenerated Protein Fibres. Courtaulds.

The wet strength of casein fibres is improved by treating the coagulated protein in methyl or ethyl alcohol, an esterification eatalyst, e.g. sulphuric acid and a phenol, and then hardening in formalin and sodium sulphate.

Acetate Fibres having Affinity for Acid Dyes. Eastman Kodak Co. USP 2,675,360 Incorporation in a cellulose ester spinning dope of a linear polyurethane containing tertiary amine groups imparts affinity for acid dyes to the fibres produced from it.

Resinated Hydrolysed Dextran Monofilaments. Commonwealth Engineering Co. USP 2,674,517
A dextran prepared by the action of micro organisms on a suitable culture medium is precipitated from the culture by adding alcohol or acetone. It is then redissolved and hydrolysed with acid and the solution fractionated by treatment with isopropyl alcohol to remove the high mol. wt. components and leave a dextran of mol. wt. 50,000–3,000,000. To this solution of dextran is added a urea-or melamine-formaldehyde resin. The resulting viscous mass is then extruded into a liquid which polymerises the resin, e.g. acetone, alcohol or 0-1x-acetic acid.

Solvent for Acrylonitrile Polymers. C. O. C. Chemstrand BP 726,685-6

Hydrolysis of NN-dimethylacetamide used as a solvent for acrylonitrile polymers cannot be prevented under commercial working conditions. However, so long as the acetic acid content is kept within 1-4% by weight good commercial products, e.g. fibres, can be obtained from the colution.

Elastic Yarns from Silicone Rubbers. Dow Corning Ltd. BP 726,479

Elastic core yarns in which the core is unaffected by perspiration, detergents, high temp, drying, and bleaching, and which retains its elasticity under conditions most detrimental to both natural and synthetic organic rubbers, are made by using an elastic filament of vulcanised silicone rubber.

4-Alkoxy-2-hydroxybenzophenones for stabilising Organic Polymers to Ultraviolet Radiation (III p. 354). Source of Unbleachable Colour in Cotton (VII p. 362). Removing Hair and Wool from Animal Hides and Skins (XII p. 366).

# VII— DESIZING; SCOURING; CARBONISING; BLEACHING

- Molecular Structure and Emulsion Stability of Aliphatic Alcohols and Esters. H. Kazi and C. M. Desai. J. Indian Chem. Soc., 31, 765-768 (Oct. 1954).
- Interfacial Tension of Aliphatic Alcohols and Esters with Surface-active Agents: Minima and Maxima in Interfacial Tension Curves. H. Kazi and C. M. Desai. J. Indian Chem. Soc., 31, 769-772 (Oct. 1954).

Influence of the Ionic Character of Surface-active Compounds in Wet Processing. W. Melliand Textilber., 36, 166-173 (Feb. 1955).

The negative charge of water is strongly increased by anion-active compounds, little changed by non-ionic compounds, and markedly diminished by cation-active compounds, so that at low concentrations of the latter a positive charge develops. In washing, anionics increase the negative charges on fibres and soil and intensify the cleaning effect. In some cases, cationics can cause inversion of the washing process. The action of non-ionics cannot readily be explained in terms of charge. The latter is also shown to play an important part in dyeing, delustring, and other operations 8. R. C.

Adsorption and Deposition of Carbon on Cotton from Solutions of Sodium Dodecyl Sulphate. R. D. Vold and A. K. Phansalkar. Roc. Trav. chim., 74, 41-51 (Jan. 1955).

The deposition of carbon on cotton cloth from solutions of sodium dodecyl sulphate of varying concentrations has been determined by measurement of surface together with the adsorption isotherms of reflectance. sodium dodecyl sulphate on both cotton and carbon and the aggregate size of carbon in the suspension. Minimum deposition occurs at a concentration of sodium dodecyl sulphate markedly below the critical micellar concentration and not correlated with either the assumed ζ-potential or the surface coverage of the adsorbed detergent.

Action of Oxidising Detergents on Cotton Cloth.

H. Krässig. Melliand Textilber., 36, 55-58 (Jan.), 163-166 (Feb.), 265-267 (March 1955).

Oxidising detergents should not contain > approx. 1.5% active oxygen, i.e. 15% NaBO4,H2O,3H4O, and products stronger than this are considered to be bleaches. The influences of potentially acid compounds and The influences of potentially acid compounds and stabilisers are discussed and the fibre damage due to molecular degradation is related to changes in morphology.

Dry-cleaning Techniques and the Work of the AATCC Committee on Dry-cleaning Test Methods. A. E. Johnson. Amer. Dyestuff Rep., 44, P108-P111 (14 Feb. 1955).

General techniques employed commercially by drycleaners in the U.S.A. are explained, with a discussion on the accomplishments of the AATCC Committee in correlating test methods with practical dry-cleaning. J. W. B.

Continuous Bleaching by Sodium Hypochlorite. R. G. Manudhane. Indian Textile J., 65, 228-229 (Jan. 1955).

Source of Unbleachable Colour in Cotton. S. A. Simon and C. Harmon. Amer. Dyestuff Rep., 44, P100-P104 (14 Feb. 1955).

Sources of colour in cotton are classified as-dirt, which is readily removed by mechanical cleaning; geo-metric, which refers to physical factors of shape; intrinsic, referring to genetic colour dependent upon seed variety; and extrinsic, which is brought about by exposure to the environment and is not mechanically cleanable. the last interferes with bleaching, and spectrophotometric evidence is presented which shows that the unbleachable colouring matter is developed by fungi. J. W. B.

#### PATENTS

#### Desizing Glass Fibre Fabric. Glass Fibers

USP 2,674,548 The fabric is treated with alkali at pH 11-13, washed to remove excess alkali and then heated for approx. I min. at 1000-1100°r, to burn the size from the fabric.

USP 2,674,549 The fabric is treated with a strong oxidising agent and then heated to burn off the size, e.g. the fabric is treated with aq. 3-10% KClO<sub>3</sub> and heated for 30 sec. at 650-750°F.

Bleaching Textiles with Ozone. H. Locali. BP 727,771
The bleaching action of ozone is much speeded up if the operation is carried out at < 40°c. C. O. C.

Bleaching Textiles other than Wool with Permonosulphuric Acid or its Salts. Stevens BP 727,419

Textiles not containing wool are bleached with aqueous permonosulphuric acid or permonosulphate at > 80°c., protein or protein-like fibres being best bleached at pH 7 while natural cellulosic fibres are best treated at pH . C. O. C.

Dependence of the Solubilising Powers of Sodium Oleate Hydrosols on their Structures (III p. 353).

Rate of Flow of Liquids into Capillaries under the Action of Surface Forces (VIII below).

#### VIII- DYEING

- Control of Quality in Dyeing and Finishing. E. Padgott. J. Textile Inst., 46, P209-P219 (Feb. 1955).
- Albert Scheurer (1848-1924). 20, 201-204 (March 1955). C. Zuber. Teintex,

20, 201-208 (march 1999).

A short biography, listing Scheurer's varied and numerous contributions to textile chemistry and his connections with Alsace and the Société Industrielle de Mulhouse. 8. R. C.

Rate of Flow of Liquids into Capillaries under the Action of Surface Forces. G. F. N. Calderwood and E. W. J. Mardles. J. Textile Inst., 46, 1161-1170 (March 1955).

Rates of penetration of liquids into capillary tubes are shown not to be in close agreement with published flow equations which take account only of the capillary, viscous, gravitational, and inertia forces. Rates of peneration are less than predicted, particularly in dry tubes; movement under small forces is jerky and often rhythmic in recession, the differences from calculated values being especially pronounced with water, formamide, cyclohexanol or benzene. Hysteresis of the equilibrium position after capillary ascent and recession respectively is studied by using capillaries inclined at a small angle from the horizontal. It is found that the hysteresis is associated with the presence of thin elastic films on the walls of the capillaries, adjacent to, and forming an integral part of, the meniscus. Differences of the contact angles and of their cosines during advance and recessional flow were found to be inadequate to explain the dynamic phenomena observed; the initial rates of penetration appear explicable by flow reversal at the meniscus together with some turbulence and by the energy involved in setting in motion the liquid exterior to the capillary. J. W. B.

Air Flow through Plugs of Textile Fibres. I— General Flow Relations. E. Lord. J. Textile Inst., 46, T191-T213 (March 1955).

Air flow through plugs of textile fibres is studied, using 44 samples of cotton, 8 of viscose rayon, 3 of wool, and 2 each of cuprammonium rayon and silk. It is found that the magnitude of the porosity effect differs from that predicted by various hydrodynamical theories, and that the nature of the fibre surface affects the flow. An empirical relation between flow and the various plug characteristics, viz. specific surface, density, and poresity, is developed; but it is shown that, if specific fibre surface is to be predicted accurately from permeability measurements, it is necessary for the flow apparatus to be empirically calibrated under conditions of use. J. W. B.

High-temperature Dycing of Dacron-Wool Blends.

Anon. Modern Textiles, 36, 43 and 88 (Jan. 1955).

Procedures are described for single- and two-bath pressure dycing of wool-Dacron blends, using a new method in which a small amount of formalin is added to the dyebath to prevent degradation of the wool. J. W. B.

Pigmented Resin Emulsions for Printing or Colouring Textiles (IX p. 363).

#### IX-PRINTING

New Developments in Vat-dye Printing. A. K. Saville. Amer. Dyestuff Rep., 44, P105-P107 (14 Feb. 1955). Methods whereby vat dyes are printed from neutral vehicles and developed by impregnating the fabric with

a caustic alkali-hydrosulphite reducing system and continuously steaming are described, and the influence of various impregnation techniques, time, and conditions J. W. B. of steaming is discussed.

Use of Alginates in Printing. C. Weimann. 20, 189-198 (March 1955). Various applications are given together with recipes.

Organic Dyes in Photography. L. Roosens. Ind. chem. Belge, 19, 1034–1050 (1954). Review, 279 references. C. O. C.

#### Prints and Colourings on Textiles. BASF.

BP 727,598 Prints and colourings are produced on fibrous materials by applying a pigmented dispersion or solution of a hydrophilic water soluble or water dispersible quaternary polyammonium salt of a polyner derived from an ethylenically unsaturated compound, e.g. pyridinium salt of a methyl methacrylate-vinyl- $\beta$ -chloroethyl ester copolymer, and then baking to split the quaternary ammonium salt.

#### C. O. C. Pigmented Resin Emulsions for Printing or Colouring BP 727,650 Textiles. FBy.

An aqueous emulsion containing a pigment, a thickener, and a copolymer of a vinyl monomer yielding a filmforming polymer if polymerised alone, e.g. methyl methacrylate, with less than its own weight of vinyl chloride, acrylonitrile or other viny! monomer acting as a cross linking agent is applied and the print heated or steamed in known manner. The resulting prints or colourings are clear, and of good fastness to rubbing, washing, perspiration and wet processing.

C. O. C.

One Component Diazotype Material. Fabriek L. Van der Grinten. Chemische

A one component diazotype material contains at least two different types of light sensitive diazo compounds and has a total content of < 0.3 millimol. diazo compound per sq.m. At least one of the diazo compounds must be of formula-

(R<sup>1</sup> and R<sup>2</sup> = Alk; X = anion and Y = a substituent having for light of 4050a. an epsilon value  $> 1.5 \times 10^4$ and for light of 3650A. one which is less than the former) e.g. stannic chloride double salt of 1-diazo-4-ethylethanolamino-3-chlorobenzene; and a second diazo compound having for light of 3650a. an epsilon value > 1.5 × 10<sup>4</sup> and for light of 4050a. an opsilon value lower than the former), e.g. 1-diazo-4-monocyclohexylamino-3-chloro-C. O. C. benzene

Heat-sensitive Record Material. National Cash Register Co. USP 2,675,332 Paper impregnated first with a mild alkali then with an eosin compound of formula-

(R = CH<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>, or C<sub>3</sub>H<sub>7</sub>) is slightly tinted yellow but on heating to 290°F. becomes deep red.

Quaternary Ammonium Salts of Formaldehyde Sulphoxy-lie Acid (III p. 363). Transfer Paper and Dry-Strip Transfers made with such Paper (XI p. 365).

#### X-SIZING AND FINISHING

Systematic Study of Sizing. V- Reagent and Method of Staining for Sizes not based on Starch.
R. Schutz and S. Marguier. Bull. Inst. Text. France, (51), 17-22 (Feb. 1955).

The yarns to be examined are treated with a solution of 1 ml. cone. HCl in 50 ml. methanol and then stained with a solution of 2.5 g. Artisil Direct Violet BP (8) and 0.5 g. Paris Violet Base in 50 ml. methanol. The yarns are sectioned and examined microscopically. Sizes containing alginate, cellulose ether, carboxymethylcellulose, polyvinyl alcohol, and polymethacylate are stained black.

9 photomicrographs are given.

J. C. F.

Influence on Quality of Steam Content in Driers. A. Meier-Windhorst. Melliand Textilber., 36, 276-280 (March 1955).

Physical and chemical changes in the goods can be detected resulting from changes in the moisture content 8. R. C.

Trade-named Finishes. M. Newman. Amer. Dyestuff Rep., 44, P138-P140 (28 Feb. 1955).

A few examples of trade-named finishes are presented, with an account of what they stand for (but not necessarily of their composition); some information is given of the tests which the finishes are supposed to pass. J. W. B.

Finishing of Cotton Fabrics. G. M. Ganta. Canadian Textile J., 66, 55-65 (25 Feb. 1955). A review of fabric preparation, finishes affecting

appearance, finishes affecting handle, and finishes affecting riceability.

Mechanism of Imparting Wrinkle Recovery to Cellulosic Fabrics. T. F. Cooke, J. H. Dusenbury, R. H. Kienle, and E. E. Lineken. Text. Research J., 24, 1015-1036 (Dec. 1954).

Cotton and viscose rayon fabrics treated with typical resin finishes have been tested for wrinkle resistance. Tests applied include wear, washing, fabric elasticity, strength and tenaile tests. Melamine-formaldehyde + styrene-ethylacrylate and melamine-formaldehyde finishes give the best recovery. Wrinkle recovery and elasticity of fabric appear to be linearly related. Chemical aspects and the results of X-ray and infrared studies are discussed, It is suggested that the attainment of wrinkle resistance is due to an increase in the elasticity of fibres. The ability of a fabric to recover from creasing is more important than its crease resistance. It is suggested that the small increase in wrinkle resistance produced by application of elastomer-type thermoplastic resins is also caused by increased elasticity of fabric resulting from elastic yarnto-yarn bridges and/or from a decrease in coefficient of friction of yarn. Possible interactions of resins of the melamine- or urea-formaldehyde type with the fibre include (1) chemical reaction between resin and cellulose, (2) resin formation, (3) hydrogen bonding and van der Waals forces between resin and cellulose. Possibly all these occur, but it is suggested that wrinkle resistance is primarily due to chemical reaction of the resin with the fibre to bridge or cross-link chains, preventing irreversible slippage of adjacent chains and reducing permanent set.

Effect of Various Softeners on a Resin-treated Viscose Rayon Fabric. AATCC Piedmont Section. Amer. Dyestuff Rep., 44, P141-P150 (28 Feb. 1955). Viscose-rayon staple-fibre gabardine shirting is treated in the state of the section of the state of the section of t with urea-formaldehyde reain followed by non-ionic, anionic, cationic, and pyridine-type softeners, each at several concentrations. The softening effect is calculated both before and after laundering. While it is not possible to make broad generalisations regarding different classes on the basis of one member of a given class, some interesting differences are found. Only the cationic and pyridine softeners produce major changes in handle, and up to 4% of the pyridine type is necessary for reduction in stiffness. Sewability and tear strengths are increased by all types, but only the pyridine type produces marked increase in wrinkle resistance before laundering. All improve it slightly after laundering. Flat abrasion resistance is reduced, but flex abrasion resistance varies Coefficient of friction is reduced, but tensile strength is not significantly affected. J. W. B.

Wool Protein free from Disulphide Cross-links. I. J. O'Donnell. Text. Research J., 24, 1058-1063 (Dec. 1954).

Modified wools have been prepared in which all the disulphide bonds have been severed by reduction with sodium thioglycollate and methylation under conditions not causing breakage of peptide links. The possibility of introducing strongly lyophilic groups into the wool protein has been investigated. Solubility, the effect of detergents, electrophoresis, and methylation of S-carboxamidomethyl-wool and extracts of it have been studied. WRM

Protection of Textiles against Microbiological Attack.

Test Methods—I. H. M. Ulrich. Textil-Praxis,
10, 187-189 (Feb. 1955).

The liabilities of various types of natural and synthetic fibres to microbiological attack are compared and various methods of protection against this attack are reviewed. The principal requirements of a protective agent are

enumerated.

Finishing the Newer Synthetic Fibres. H. Moore.

Amer. Dyestuff Rep., 44, P133-P136 (28 Feb. 1955).

A brief survey of finishing methods for nylon, Dacron,

J. W. B.

Reducing the Felting Power of Wool. British Industrial RP 727 660

Wool, if desired in presence of other fibres, is treated with an acidic aqueous true solution of the components of an aminoplast resin and/or a precondensate of such a resin at 80-100°c. until the requisite amount of resin has been absorbed, after which excess liquor is removed and the material dried without being rinsed.

Flameproofing Lightweight Cellulosic Materials BP 727,700

The material is treated with an aqueous solution containing HCl and a titanium chloride acylate. There containing flot and a training chorace asystes. These should be 10.5 g./l. Ti and 1-3 acylate groups per atom of Ti. The total chlorine present should be 90-200 g./l. The effect may be increased by adding SbCl<sub>3</sub> to the soln. in the proportion of 1.7-3.4 parts 8b for each part of Ti, the chlorine of the SbCl<sub>3</sub> being additional to the 90-200 g./l. mentioned above.

Créping. T. Ambrus. Moist paper or fabric is interlayed with a fine sponge like substance, e.g. orêpe rubber, and then, while the layers are firmly held together, the assembly is compressed widthways or lengthways. Thus the material may be fed between two endless crêpe rubber belts which are pressed together over a portion of their length to form the assembly.

C. O. C. Water Repellent Finish. Ciba. BP 727,015
The material is treated with a solution in an organic BP 727,015 solvent of (1) a water-insoluble methylolaminotriazine derivative which may be partially precondensed to a resinous product and which contains bound to the O atom of a methylol group at least one aliphatic residue of > 3 C. and (2) a water-repellent wax-like compound, e.g. paraffin wax, and then, after removal of the solvent, baked at 125-150°c. It is better if the solution contains an acid or

potentially acid catalyst.

Bonding Vinyl Chloride Polymers to Regenerated Cellulose Fibres, BrC.

Bonding of polyvinyl chloride and similar polymers to high tenacity, regenerated cellulose fibres or those composed of a linear polymer containing recurring units made up of C, H and an element more electronegative than C, is greatly facilitated if the fibres are first coated with an aldehyde-phenol condensate (molecular proportion 1:2 to 1.1:1).

Flame-resistance and Waterproof Brattice Cloth.
Commercial Plastics. BP 727,163

A flame-resistant and waterproof brattice particularly suitable as fireproof curtains in coal mines, consists of a woven fabric which has been first impregnated with a non-deliquescent flameproofing agent, e.g. ammonium phosphate and/or ammonium tungstate, and then coated on both sides with a flexible waterproofing polyvinyl chloride or vinyl chloride-vinylidene chloride copolymer composition containing a flame-resisting plasticiser, e.g. tricresyl phosphate and, preferably, an afterglow suppressent, e.g.  $Sb_2O_4$ . C. O. C.

Coated, Flexible Sheet Materials. FBy. BP 727,321 Lightweight coated fabrics of good elasticity and handle are obtained by coating a flexible sheet with a porous blown layer prepared from condensates of polyesters and di- or tri-isocyanates, e.g. that obtained by treating the polyester of 3 mol. adipic acid, I mol. hexanetriol and 3 mol. butylene glycol with toluylene dissocyanate. C. O. C.

Coating Nylon with Styrene-Acrylonitrile Copoly-mers. United States Rubber Co. USP 2.674.547 The adhesion of styrene-acrylonitrile copolymers for nylon is greatly improved by adding to the coating composition a potentially resin-forming phenol-alcohol, 2:6-dimethylol-p-tert.butyl phenol, and heating the coated nylon to condense the phenol. C. O. C.

BrC. knitted fabric is coated with a tacky coating of an adhesive which can be hardened to give a non-tacky film of rubber-like elasticity. Fibres are then projected with their axes perpendicular to the adhesive surface after which the adhesive is hardened. This yields pile fabrics which can be readily moulded to shape, e.g. for shoe uppers, gloves, etc.

Static Electricity in Textiles (VI p. 360). Control of Quality in Dyeing and Finishing (VIII p. 362).

#### XI- PAPER AND OTHER CELLULOSIC **PRODUCTS**

Effect of Molecular Weight on the Transition Temperatures of Cellulose Nitrate. P. V. Kozlov and E. F. Russkova. Dokladý Akad. Nauk S.S.S.R., 99, 105-108 (1 Nov. 1954).

Investigations previously reported (J.S.D.C., 70, 595 (1954)) on the changes of state occurring in plasticised cellulose nitrate of mol. wt. 115,000 are extended to cellulose nitrate preparations varying in mol. wt. M from 26,000 to 408,000. When not plasticised and when plasticised with non-compatible plasticisers, these polymers remain in the vitreous state over the whole of the temp, range investigated (-80° to +140°c.), the case temp. range investigated ( $-50^{\circ}$  to  $+140^{\circ}$ ), the case of deformation, however, becoming greater and increasing more rapidly with rise in temp. as M increases. Addition of dibutyl phthalate (0.75 mol. per glucose residue) results in a change of state at  $25-30^{\circ}$ c., irrespective of the value of M. When M < 100,000, the change is from the vitreous state to one of viscous flow; but when M > 100,000, the second state is rubbery and a further change to the viscous-flow state occurs at a still higher temp., which rises as M increases and is  $130^{\circ}$ c, when M = 408,000.

Molecular Weight Distribution of Benzylcellulose. H. B. Roy. J. Indian Chem. Soc., 31, 677-682 (Sept.

The mol. wt. distribution of benzyloelluloses, prepared (a) by benzylating sodium cellulose three times with benzyl chloride in NaOH and (b) benzylating sodium cellulose once only in toluene, was determined by fractional precipitation from chloroform solution with ethanol and measurement of the intrinsic viscosity of the fractions. Fractionation was shown to be sharp by turbidimetric titration. Analysis of a number of fractions indicated a fairly uniform chemical composition. Differential mol. wt. distribution curves for the two ethers differ in the position of the maximum (mol. wt. 5,000 for (a) and mol. wt. 28,000 for (b)), but have the same general shape. Variations in the benzylating conditions for (b) produce marked changes in the differential distribution curve. Toluene as solvent gives a single max. at mol. wt. ca. 15,000; with no solvent, two max, are obtained at mol. wt. 9,500 and 24,500; with toluene in a nitrogen atmosphere the curve shows maxima at mol. wt. 9,500, 20,000, and > 35,000 (uncompleted). Although the unfractionated material produced in  $N_1$  is completely sol. in chloroform, the first two fractions obtained from it (high mol. wt.) are not. The shape of the differential distribution curve changes when the fractionation is carried out from a benzene-alcohol mixture with petroleum ether. A. J.

by primary valencies.

#### Anomalous Behaviour of Benzylcellulose prepared from Egyptian Cotton. H. B. Roy. J. Indian Chem. Soc., 31, 721-725 (Oct. 1954).

Intrinsic viscosity measurements have been made on bensylcellulose fractions precipitated from chloroform solution by the addition of ethanol. The precipitation was irregular, and the order of mol. wt. reversed at some stages. Mol. wt. calculated from viscosity measurements are much higher than those obtained from camotic pressure. This is explained by assuming cross-linking

#### PATENTS

Modified Paper, Cluett, Peabody & Co. BP 727,261
Stretchable paper having smooth substantially parallel surfaces and an extensibility permitting a permanent elongation in excess of the normal elasticity without decrease in thickness is made by pushing and crowding the fibre together in a direction opposite to the desired extension, while the web is in a plastic condition, and preventing créping by applying pressure on the web normal to its surfaces. A suitable plastic condition is attained by removing only part of the moisture as the web leaves the wet end. The pressure on and contraction of the web are produced by pressing between two surfaces, one heated and having relatively low frictional resistance to the web, and the other smooth but having a relatively high frictional resistance to the veb and consisting of rubber or other contractional material. The contraction process may be repeated several times.

8. V. S.

Coating Webs. Kodak.

A thin set layer from a settable composition is formed on a flexible supporting layer by depositing an aqueous solution of the composition and a spreading agent in the form of a thin liquid coating on a rigid surface, setting the coating, pressing the wet layer and the supporting layers to bond them and stripping the laminated layers from the rigid surface, which may comprise a rotatable drum. The thin layer may be set by change in temperature or by chemical means, e.g. polyvinyl alcohol containing borax may be set by fuming with ammonia gas. The rigid surface may be provided with a fatty acid surface to facilitate the stripping of the set layer.

8. V. S.

Transfer Paper and Dry-Strip Transfers made with such Paper. Brittains. BP 727,825 An improved transfer paper comprises a backing paper that will not delaminate in use united by a mechanically rupturable bond to a relatively thinner tissue paper which is permanently coated with a carrier film of translucent flexible material adapted to receive and form the desired

Transfer Paper and Dry-Strip Transfers made with such Paper. Brittains. BP 728,089
A transfer comprises a backing paper strong enough

A transfer comprises a backing paper strong enough to withstand delamination in use united to a carrier film of translucent flexible material which is united in turn to a transfer film. Both bonds are mechanically rupturable, and the carrier film may consist of polyethylene or polyethorotrifluoroethylene applied to the paper by extrusion lamination.

8. V. S.

Antitaraish Paper. Dominion Cellulose. BP 726,803
The process comprises adding a soluble salt of copper, lead or zine to the pulp in the beater, and precipitating the insoluble basic carbonate of the respective metal on the pulp fibres in a finely divided form by the addition of a soluble carbonate, e.g. Na<sub>5</sub>CO<sub>3</sub>, to bring the pH of the stock to at least 6·0. A soluble cadmium salt may be used, the insoluble normal carbonate being precipitated, and enough metal salt is used in each case to give 0·1 to 4·0% precipitate, calculated as metal, on the weight of the paper. A soluble salt of rosin acid, e.g. the sodium salt, or a soluble carboxymethyl cellulose salt, e.g. the sodium salt, may be added to the beater stock before the soluble metal salt is added.

8. V. S.

# Controlling the Degree of Adhesion of Polyethylene Coatings to Paper. Union Carbide and Carbon BP 728,203

Adhesion of extruded polyethylene coatings calendered on to paper is improved by heating the coated paper to 130-175°c. for >> 10 sec., or to 110-150°c. for >> 8 sec.

if the polyethylene is mixed with paraffin or microcrystalline wax. The coated paper may be passed over a revolving drum beneath a bank of radiant heaters, the drum being heated to 105-195°c.

Photo-Sensitive Diazotype Coated Paper Plate for Photo-Lithography. S. D. Warren Co. BP 728,205 A photo-sensitive coated paper product, capable of being transformed by actinic light into a planographic printing plate comprises a paper base having a calendered coating layer comprising an insolubilised carbalkoxyhydroxyalkyl cellulose adhesive and a greater quantity of a non-hydrous pigment, e.g. blane fixe or titanium dioxide, and having over the coating a photosensitive layer comprising (1) a diazonium sait of formula—

(X = anion; Ar = phenylene residue which may be substituted by alkyl, alkoxy, aryloxy, arylsulphamido groups and/or halogen; R¹ = Ar which may be substituted by alkyl, alkoxy, earboxy groups and/or halogen; R² = H or Alk), or (2) a condensate of such diazonium salt with aldehydes, or (3) a sulphonate obtained by reacting (1) or (2) with a sulphite. The adhesive may be sodium carboxymethylhydroxyethyl cellulose insolubilised by an amino-rosin, e.g. acidified melamine-formaldehyde condensate. On one side of the paper base may be a barrier coat an anchoring coat comprising non-hydrous pigment and insolubilised protein adhesive and over the anchoring coat the calendered hydrophilic coating and the photosensitive layers mentioned above. The photosensitive may be made by the action of a sulphite on the formaldehyde condensate of a paradiazonium salt of a diphenylamine.

3. V. 8.

4-Alkoxy-2-hydroxybenzophenones for stabilising Organic Polymers to Ultraviolet Radiation (III p. 354).

Affinity of Black Wattle Extract Constituents. I—
Affinity of Polyphenols for Swollen Collagen and Cellulose in Water (XII p. 366).

#### XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Fastness of Dyes on Leather. F. P. Russell and T. C. Mullen. J. Soc. Leather Trades Chem., 39, 70-80 (March 1955).

Examination of the effects of tannage, dyes, levelling agents, cationic fixing agents, and dyeing methods on the fastness properties of dyed leather, with special reference to light, washing, and perspiration, shows that, owing to the number of factors involved, no predictions can be made. Each case must be considered on its own merits, and the results depend equally upon the methods of tanning and the dyes employed. The need for accepted standards of fastness (other than that to light—B.S. 1006 is referred to) relating to the dyed leather, and not to the dyes themselves, is urged. A large amount of experimental evidence is tabulated and discussed.

J. W. D.

Influence of Acidity on the Colour of Vegetable Tannin and Leather. G. A. Bravo, Oct. Leder-Z. Festnummer, B 24 (1954): J. Soc. Leather Trades' Chem., 39, 99 (March 1955).

Colours of solutions of chestnut, sumae, and quebrache have been compared by Tintometer and Beckmann spectrometer; the greatest deviation is found in the red region. The source of light is most important in colour determination. The colour is given of a series of chestnut liquors of pH from 2-0 to 7-2, with the corresponding colour of the leathers produced therefrom. In evaluating colour, the blue value is multiplied by 3, the red by 2, and the yellow by 1; the "total colour" is the sum of the three values. It is found that the chestnut liquor at pH 2-0 has the lightest colour, while leather of the lightest colour is produced from a liquor of pH 4-8. Good colour results are obtained when chestnut liquors are adjusted with phosphoric acid and disodium phosphate.

C. J. W. H.

Salt Stains, Bacterial Stains. W. Hausam. 247 (1954); A. Kuntzel and T. Stirts. III m. Loder, 5, Ibid., 5, 247 (1954): J. Soc. Leather Trades' Chem., 39, 98 (March 1955).

Bacteria have often been isolated from salt stains on both the flesh and the grain sides of skins. The theory that salt stains are caused chemically by impurities in curing salt is disputed and evidence is given to show that they are of bacterial origin.

In a reply to W. Hausam, the latter two authors main-tain that the evidence of the chemical origin of salt stains is conclusive. C. J. W. H.

Iron Stains in Vegetable Tanning. H. Grunewald. Osterr, Leder-Ztg., Festival No., D16-23 (1954): Osterr. Leder-Ztg., Festival No., D. Chem. Abs., 49, 4316 (25 March 1955).

Generally speaking, synthetic tanning agents are less sensitive to iron than natural ones. Sensitivity is lower at sensitive to iron than natural ones. Sensitivity is lower at higher pH and is further diminished by pretanning. Of the chelating agents used for preventing precipitation of Fe by tanning agents, the polyphosphates, especially  $(NaPO_3)_{ab}$ .  $Na_4P_3O_{10}$ , and  $Na_4P_3O_{10}$ , are the best, being useful for removing Fe from tanning liquors and also, if 3% is added to the tanning liquor, for bleaching tanned leather discoloured by iron compounds and for removing C. O. C.

Development of Zirconium Tannage. M. Paquet. Octerr, Leder-Zig., Festival No., D6-9 (1954); Chem. Abs., 49, 4315 (25 March 1955).

Difficulties in tanning with ZrO2 are mainly caused by the low pH (< 1.8) of the tanning extracts. Better results can be obtained by using Zr salts of organic acids, especially acetic but not formic acid, instead of salts of mineral acid, and by keeping the extracts at pH 2.6-3.0. The amount of water should be 160-180% on the weight of the unhaired hide, and the ZrO<sub>2</sub>: acid ratio (acid calculated as H<sub>2</sub>SO<sub>4</sub>) should be 0.65–0.70. 3–4% of the Zr compound on the weight of the hide should be used.

Collagen Pores determined by Electron Microscopy. M. Swerdlow and R. R. Stromberg. Bur. Stand. J. Res., 54, 83-90 (Feb. 1955).

The existence of pores of the order of 150 A. radius within collagen fibres is demonstrated. Structural features of collagen impregnated with mercury under pressure are compared with those in the absence of mercury. A helical configuration of subfibrillar elements is suggested. Results are in agreement with conclusions regarding the presence and probable size of small pores deduced theoretically and provide information regarding the shape, location, and arrangement of nores.

W. R. M. and arrangement of pores.

Infrared Spectrum and Structure of Collagen.
R. M. Badger and A. D. E. Pullin. J. Chem. Phys.,
22, 1142 (1954): J. Soc. Leather Trades' Chem., 39, 65 (Feb. 1955).

From spectrographic evidence, it is unlikely (i) that all, or even the greater part of the peptide bonds are in the cis configuration; and (ii) that the eight-membered ring occurs to a significant extent in collagen.

C. J. W. H.

Function of Hydroxyproline in Collagens. Gustavson. Nature, 175, 70-74 (8 Jan. 1955).

The structural stability of collagen is considered to derive in part from hydrogen bonds formed between CO-NH groups, and OH groups furnished by hydroxyproline residues; this accords with the fact that stabilit increases with content of hydroxyproline. It is probable that only a few of the CO-NH and OH groups participate in interchain cross-linking. The theory is discussed at length, in terms of the hydrothermal stability of collagen and its affinity for reagents such as Cr cpd., tannins, and polyphenols. The strong bond which is the mainstay of collagens in general is probably an ester linkage; the origin of the OH group that is involved is not known. There is liberal reference to other work in this field, and 47 references are made to the literature.

Affinity of Black Wattle Extract Constituents. I-

Affinity of Black Wattle Extract Constituents. 1—Affinity of Polyphenois for Swollen Collagen and Cellulose in Water. D. G. Roux. J. Soc. Leather Trades Chern., 39, 80-91 (March 1955).

The affinity of various polyphenois for swollen collagen has been studied by a combination of column (collagen) and two-dimensional paper chromatography. With the exception of flavonois, a parallel exists between the affinity of the collagen and for collulose in of various polyphenois for collagen and for cellulose in Paper chromatograms may thus be used water. Faper chromatograms analy thus to desire to interpret differences and changes in the affinity of the major black wattle polyphenols towards collagen. The anomalous behaviour of flavonols, due to their abnormally high affinity for cellulose, is examined. The affinity of the individual polyphenols for collagen varies enormously and depends upon a combination of various factors such as solubility in water, spatial orientation, mol. size, and the number and the position of functional groups. The affinity properties are considered to operate even under the very altered conditions of practical tannage.

Anhydrous Hydrogen Fluoride as a Solvent for Collagen. R. E. Dreikom and V. O'Gorman. Nature, 175, 385 (26 Feb. 1955). Anhydrous HF dissolves steer hide collagen, but as

characteristic collagen fibrils cannot be regenerated from the soln., it is considered that denaturation accomregenerated J. W. D. panies dissolution.

Chromium Fixation on Acetylated Collagen. R. L. Sykes. J. Soc. Leather Trades Chem., 39, 56-61 (Feb. 1955).

Tannages of normal, acetyl, and esterified collagen with cationic Cr<sub>3</sub>(SO<sub>4</sub>)<sub>3</sub> suggest that two reactions occur during chrome tanning. One involves the formation of co-ordination linkages between the Cr complex and the COOH groups of the collagen, and bifunctional (but not unifunctional) linking enhances hydrothermal stut not unifunctional of the college, and bifunctional co-ordination probably decreases with increasing Cr fixation, thus accounting to some extent for the logarithmic relationship between chrome content and shrinkage temp. The second reaction is that of the OH groups of the collagen with Cr; this confers enhanced resistance to exhaustive washing, but does not improve hydrothermal stability. In normal Cr2(SO4)3 tannages this second reaction plays a minor J. W. D.

Collageneous Changes in the Intervertebral Disc with Age. A. Naylor, F. Happey, and T. Maerae. Brit. Med. J., No. 4887, 570 (1954): J. Soc. Leather Trades Chem., 39, 63 (Feb. 1955).

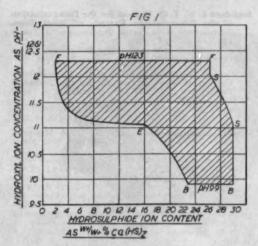
X-Ray diffraction diagrams show that the collagen of the intervertebral disc, with age, becomes increasingly oriented; this results in loss of gel structure and impaired elasticity.

Optical Rotation and Polypeptide Chain Configura-tion in Proteins. C. Cohen. Nature, 175, 129-130 (15 Jan. 1955).

Randomised chain configurations, provided by condi-tions which cause "denaturation", exhibit rotations believed to correspond to the sums of effectively independent contributions of the L-amino acid residues present The rotation of the native state appears to be the result of effects of specific main-chain configuration superimposed or effects of specific main-chair configuration superinsposes on or otherwise changing the optical rotation from that of independent, asymmetric a-carbon atoms alone. The resultant total rotation of the native protein may be appreciably greater or less than that obtained after denaturation; examples of the two possibilities are discussed in detail. Pyrrolidine residues may be influential in determining optical rotation. J. W. D.

Removing Hair and Wool from Animal Hides and Skins. H. Zaleman.

BP 726,842
A depilating paint consists of an aqueous composition containing an oxide or hydroxide of Ca, Sr, Ba, Mg and Na, and a sulphide or hydroxulphide of Ca, Mg, Sr, Al, Be, and Ne, I to not have a Waiter content of 860. Al, Ba and Na. It must have a Na ion content of 1-60% by wt. of the hydrosulphide ion content. The hydrosulphide ion contents to within the range depicted by the shaded area in the Fig.



Use of the paint avoids excessive plumping and damage to the pelt and the wool and results in easier pulling.

Moulding Skins and treating them with Liquids (I p. 353).

#### XIII— RUBBER: RESINS: PLASTICS

Thermal Degradation of Polymers as a Function of Molecular Structure, S. L. Madorsky and S. Straus. Bur. Stand. J. Res., 53, 361-370 (Dec. 1954). Vacuum pyrolysis of polymers at 200-500°c, yields

volatile fragments of varying size, depending on the nature of the polymer. Some, like polymethylene, yield only monomer. Others, like polymethylene, yield fragments varying in size from 2 C to 50 C or more. Polyisobutylene is of intermediate character, yielding monomer and large fragments. The rates of formation and volatilisation of fragments vary with different polymers. tetrafluoroethylene is thermally the most stable and poly-a-methylstyrene the least. Differences in thermal poly-a-methylstyrene the least. Differences in thermal behaviour can be correlated with molecular structure and with the nature and frequency of side groups.
W. R. M.

PATENTS

Thermoplastic Sheets and Moulded Products. Semtex BP 727,393

Loose fibres are mixed with a finely divided polyvinyl compound and a liquid plasticiser, and the mass then heated to above the temperature above which the plasticiser is absorbed by the polyvinyl compound but below that at which gelation occurs. This results in the polyvinyl compound being firmly bonded to the fibres. If a powdered filler is also mixed in, the filler becomes bonded to the This yields a loosely fibrous mass which can be carded or which can be heated under pressure to form a compacted thermoplastic composition. C. O. C.

Polyvinyi Ester Emulsion yielding Stable Films.
Vinvi Products.

BP 726,927

A monomeric vinyl ester is dispersed in an aqueous solution of a starch derivative, the starch derivative being 10-90% by wt. of the total solids. The dispersion is heated in presence of a pelymerisation catalyst, the amount of catalyst being within the ranges of 1.5-10%by wt. on the total solids and 2-25% by wt. on the vinyl ester. The resulting emulsion is stable and deposits a continuous film which is waterproof and stable to wet C. O. C.

Bismuth Oxycarbonate or Oxysalicylate for produc-ing Pearlescent Effects in Plastics. British Resin BP 727,801

Pearlescent effects are produced by adding 1-20% weight of bismuth oxysalicylate or 0.1-10% of bismuth oxycarbonate to a cellulose derivative or a polystyrene resin or 1-5% of biamuth oxycarbonate to a phenol-formaldehyde resin. C. O. C. formaldehyde resin.

Coating Transparent Plastics with Aluminium Bronze. Wilmot-Breeden. BP 726,671

A bright golden coating is produced on transparent plastics by applying the vaporised metal in a vacuum of about 0.1 micron mercury pressure after the plastic and the vacuum chamber have been cleansed by a high tension voltage electrical discharge in a vacuum of about 5-0 micron mercury pressure.

4-Alkoxy-2-hydroxybenzophenones for stabilising Organic Polymers to Ultraviolet Radiation (III p. 354). Epoxy Resin and Polyamide Compositions (III p. 354 Syntheses of Organic Fluorescent Compounds. XVII-

Syntheses of Water-soluble Fluorescent Compounds by Sulphomethylation of Monoamino Compounds and their Fluorescent Brightening Effect. XVIII—Syntheses of Water-soluble Compounds by Sulphomethylation of Diamino Compounds and their Fluorescent Brightening Effect. XIX—Syntheses of Fluorescent Compounds by Cyanomethylation and their Fluorescent Brightening Effect. XX— Syntheses of some Fluorescent Resins (IV p. 355).

#### XIV-ANALYSIS; TESTING; APPARATUS

New Reagent for the Histochemical and Chemical Detection of Calcium. 8. M. McGee-Russell. Nature, 175, 301-302 (12 Feb. 1955).

The dye "kernechtrot" (G. T. Gurr Ltd. (Batch 3569),

or T. Gerrard Ltd. (nuclear fast red standard) ) is a sensitive reagent for the detection of Ca. A saturated aq. soln. of the dye (which is first purified by washing twice with dist. water) is used, and a brilliant red lake is pptd. in the presence of Ca. The laking response is not prevented by KOH, provided the pH is not too high, but no lake is formed with highly insol. Ca salts such as the oxalate. The reagent will distinguish readily between tap water and dist. water. The name "calcium red" is proposed for the dye.

J. W. D.

Chromatographic Separation of meta- and para-Cresols. D. White and D. W. Grant. Nature, 175, 513 (19 March 1955).

m- and p-Cresols, which are normally extremely difficult to separate, may be resolved upon a column packed with "Cellite 535" containing a phosphate buffer of pH 11.5. The mobile phase (cyclohexane) is forced through the column by pressure from a nitrogen cylinder. The progress of the chromatogram is followed by a spot test which is given by 2-chloro-4-nitrobenzenediazonium naphthalene-2-sulphonate (development of red colour in NaOH soln.); this test will detect 5 p.p.m. of cresol quite readily. The ortho momer is also separated, though this normally presents little difficulty. The method has not yet been The method has not yet been established on a quant. basis.

Colour Reactions of Unsaturated Carbonyl Compounds. I—Use of Azobenzene-4-hydrazine-sulphonic Acid. S. Hünig and J. Utermann. Chem. Ber., 88, 423-429 (March 1955).

Decomposition of Azo Dyes in Acid Solution. L. 8. Harrow and J. H. Jones. J. Assocn. Off. Agric, Chem., 37, 1012-1020 (1954): Chem. Abs., 49, 2737 (25 Feb. 1955).

Decomposition in boiling 0-1n-HCl of three types of azo dyes of formula-

$$R^{1} \underbrace{\hspace{1cm} N: N- \underbrace{\hspace{1cm} R^{2}}_{\hspace{1cm}} R^{2}}$$

was investigated (Type A: R<sup>1</sup> = H or SO<sub>2</sub>H; R<sup>2</sup> = H; R<sup>3</sup> = OH or NH<sub>2</sub>; Type B: R<sup>1</sup> and R<sup>2</sup> = H; R<sup>2</sup> = OH; Type C: R<sup>2</sup> and R<sup>3</sup> = H; R<sup>3</sup> = NH<sub>2</sub>).

Type A dyes (Orange I, 1-hydroxy-4-phenylazonaphthalene, and 1-amino-4-phenylazonaphthalene) were 90% destroyed by refluxing for 1-3 hr. With Orange I the following decomposition products were identified: 1:4-parkthornizona multipartite acid 2-bydroxy 1.4. naphthoquinone, sulphanilie acid, and 2-hydroxy-1:4-naphthoquinone. With stronger solutions of Orange I,

2-p-sulphoaniline-1:4-naphthoquinone was also isolated. The decomposition is partly reversible. p-Sulphophenylhydrazine (20) with 1:4-naphthoquinone (20) yielded Orange I (2) when refluxed for 4 hr. in 0-1x-HCl. The mechanism proposed by Bucherer et al. does not account for the formation of p-sulphophenylhydrazine or 1:4-naphthoquinone. Type B dyes, e.g. Orange II, decompose more slowly but with analogous decomposition products. Of the Type C dyes, FD&C Yellow No. 3 yielded a mixture of phenol and 2-naphthylamine quantitatively. This indicates that Type C dyes decompose by reversal of the coupling mechanism and that the diazonium compound produced is further decomposed to the corresponding phenol.

C. O. C.

Detection of Vascular Tissues available for Water Transport in the Hop by Colourless Derivatives of Basic Dyes. P. W. Talboys. Nature, 175, 510 (19 March 1955).

X-Ray Diffraction Techniques appropriate to the Study of Organic Fibres. C. Legrand. Kolloid-Z., 140, 112-120 (Feb. 1955).

A method of X-ray diffraction adapted to fibre study is described, involving three complementary experimental techniques. One of these uses monochromatic radiation focussed by a curved crystal and permits precise determination of lattice variations and of crystallinity. In another, simpler method a vacuum chamber is used chiefly to study preferential orientations in the ordered regions of the fibre. Measurements of the crystalline lattice can also be made. A coaxial chamber is used to examine orientations of different crystallographic planes. Information regarding large angles is obtained while interferences corresponding to reticular distances of 50-60 A. can be registered.

W. R. M.

AATCC Rapid Control Tests—Simplified Tests for Fabric Properties. T. G. Hawley. Amer. Dyestuff Rep., 44. P114-P115 (14 Feb. 1955).

The value of simplified small-scale tests for consumer fabrics is discussed.

J. W. B.

Comparison of the Willows-Markert Swelling Reaction with the Mean Degree of Polymerisation of Cotton. C. Stingl and H. Vollenbruck. Melliand Textiber., 36, 217-218 (March 1955).

A connection exists between the above test for damage in cotton and D.P., and is illustrated photomicrographically. S. R. C.

Determination of Xylan in Jute. W. G. Macmillan, A. B. Sen Gupta, and A. S. Dutt. J. Textile Inst., 46, T214-T224 (March 1955).

Methods of estimating furfural are surveyed, and a procedure is developed in which the volumetric method of Kullgren and Tydén is modified, without using a catalyst, to make it more suitable for chemically treated jute. Pure xylose is distilled with 13·15% HCl esturated with NaCl, and sufficient 1-8v NaOH added to 50 ml. of distillate to maintain the HCl content at 1%. A 25 ml. portion is treated with bromide-bromate (0·05v.), and the unchanged Br determined. Effects of time of treatment, temperature, acid concentration, and furfural content are studied. A comparison is also made of gravimetric and volumetric methods for determining furfural. Variations in the method of distillation or estimation have no significant effect on the yield. The presence of formaldehyde does not interfere with the determination of furfural by the modified volumetric procedure, and no formaldehyde is liberated from jute lignin under the conditions of furfural distillation. The apparent yield of furfural from all varieties of jute represents not only true furfural from all varieties of jute represents not only true furfural from all varieties of jute represents not only true furfural from hexose residues. Some furfural is not recovered in the rapid distillation method (1 g. jute distilled with 210 ml. 13·15% HCl saturated with NaCl, at 25 ml. per 10 min., 130 ml. being collected), but the presence of hydroxymethylfurfural counterbalances this loss to give a correct indication of the true furfural content.

J. W. B.

Paper Chromatography in Fibre Research. D. B. Das. Indian Textile J., 65, 156-161 (Dec. 1954).

A useful review of work in connection with wool, silk, cotton, flax, and jute.

J. W. D.

Standard I. W. T. O. Method for the Determination of the Ether-soluble Extract of Wool Tops.

International Wool Textile Organisation. J. Textile Inst., 46, 845-846 (March 1955).

This method is primarily intended for tops, but is suitable for other forms.

J. W. B.

Studies on the Determination of the Ether-soluble Extract of Wool carried out by the Technical Committee of the I.W.T.O. M. Robinet. J. Textile Inst., 46, 847-850 (March 1955).

Already abstracted in J.S.D.C., 71, 71 (Jan. 1955). J. W. B.

Determination of Ardil in Ardil-Wool Mixtures.
M. V. Glynn. J. Textile Inst., 46, T228-T230 (March 1955).

Mixtures of loose wool and Ardil (ICI) top are boiled under reflux with HCl for 45 min., using 40 ml. of acid to 1 g. of fibre. Acid of 1x. cone. completely dissolves the Ardil, and permits recovery of 84% of the wool; although there is an increase in the proportionate loss of wool with increasing wool content of the mixture, results are reasonably reproducible for any given blend. Considerable variation may, however, be caused by previous processing of the wool, e.g. chlorination or bleaching.

J. W. B.

Determination of Cellulosic Constituents in Asbestos and Asbestos-Glass Textile Materials. C. Z. Carroll-Porezynski. J. Textile Inst., 46, r65-r70 (Feb. 1955).

Although cellulosic constituents in asbestos-cellulose fibre mixtures are usually determined by ignition at  $800^{\circ}$  c., it is considered that lower temperatures, e.g.  $360-383^{\circ}$  c., may be more suitable, as the loss in wt. of chrysotile asbestos is smaller, more consistent, and more independent of the time of heating than at the higher temp. A method is described in which a temp. of  $370\pm10^{\circ}$  c. is used for  $1\,\mathrm{hr}$ , the wt. of residue being divided by a correction factor of 0.975. The method is also applied to viscose rayon–glass fibre mixtures; the rayon leaves about 1% of sah and the glass loses a similar amount, the errors tending to cancel each other.

Comparison of AATCC Sunlight Tests with the Proposed International Daylight Test. AATCC Committee on Colour Fastness to Light. Amer. Dyestuff Rep., 44, P131 (28 Feb. 1955).

The I.S.O. Daylight Method gives ratings 0.4-1.7 higher than the American AATCC Sunlight Method for

The I.S.O. Daylight Method gives ratings 0·4-1·7 higher than the American AATCC Sunlight Method for samples of L2 to L6. Samples with a high fastness corresponding to L7 are rated equal. The reason is found to be the lower fastness of the Continental Blue Standards L2-L6 inclusive. J. W. B.

Flammability of Clothing Textiles. G. S. Buck.

Amer. Dyestuff Rep., 44, P115-P116 (14 Feb. 1955).

The development of the AATCC flammability test method is described to illustrate how textile testing methods can become the busis for legislation. J. W. B.

Electron Microscopy of Fibres. K. Amboss, H. W. Emerton, and J. Watts. Proc. Tech. Sects. British Faper & Board Makers' Assocn., 35, 487-510 (Dec. 1954).

By using the electron microscope as a reflection instrument for studying the effect of beating on papermaking fibres, the replication or disintegration of fibres needed when it is used as a conventional transmission instrument is eliminated. The great depth of focus and the "solid" appearance of the fibre are demonstrated, a resolution of at least 1500 a. being obtained. The shortcomings of the method are also discussed.

Action and Formulation of Antistatic Preparations (III p. 353).

Action of Acids on Cotton and Rayon (VI p. 360).

Dry-cleaning Techniques and the Work of the AATCC Committee on Dry-cleaning Test Methods (VII p. 362).

Air Flow through Plugs of Textile Fibres. I—General Flow Relations (VIII p. 362).

Fastness of Dyes on Leather (XII p. 365).





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All inquiries relating to Advertisements in the Journal should be addressed to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

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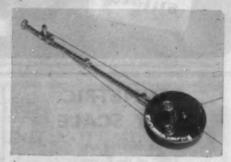
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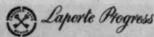
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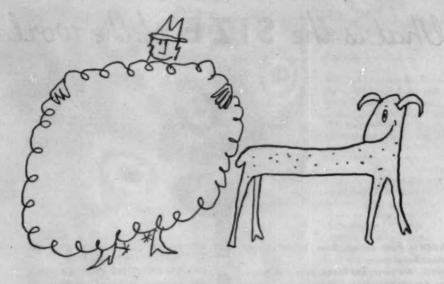
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